PELLETING AND CHARACTERIZATION OF DRY DISTILLERS' GRAIN WITH SOLUBLES PELLETS AS BIO-FUEL

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ABSTRACT

Biofuels are made from an extensive selection of fuels derived from biomass, including wood waste, agricultural wastes, and alcohol fuels. As a result of increased energy requirements, raised oil prices, and concern over greenhouse gas emissions from fossil fuels, biofuels are acquiring increased public and scientific attention. The ethanol industry is booming and during the past several years, there has been an increase in demand for fuel ethanol and use of its co-products. To increase potential revenues from ethanol processing and its utilization, extensive research is proceeding in this field. In Western Canada, wheat is the primary raw material used in the production of ethanol by fermentation and distillers' dried grains with solubles (DDGS) are one of the major co-products produced during this process. At present, the DDGS are generally sold as animal feed stock but with some alteration they could be used in other useful areas.

Densification of biomass and use of it for fuel like wood pellets, hay briquettes, etc. have been studied for many years and have also been commercialized. In this thesis, pellets made from distillers' dried grains have been investigated. DDGS were obtained from Noramera Bioenergy Corp. and Terra Grain Fuels Ltd. Before transforming them into pellets, they were characterized on the basis of physical and chemical properties. A California pilot-scale mill (with and without steam conditioning) was used for pelleting the distillers' grains with solubles.

A full factorial design with two levels of moisture content (i.e., 14 and 15.5% (w.b.)), hammer mill screen size (i.e., 3.2 and 4.8 mm) and temperature (i.e., 90 and 100°C) was used to determine the effects of these three factors on the pellet properties made from Noramera Bioenergy Corp., without steam conditioning. Different levels of moisture content were used for

the pellets made from Terra Grain Fuels Ltd. (i.e., 11.5 and 13.09% (w.b.)), with steam conditioning. The initial moisture contents of the DDGS were 12.5 and 13.75% (w.b.) from Noramera and Terra Grain, respectively. The moisture content of DDGS grinds ranged from 11.6 to 12.03% (w.b.) for the Noramera samples, and from 11.5 to 13.09% (w.b.) for Terra Grain DDGS. The moisture content decreased with a decrease in the hammer mill screen size.

The use of a smaller screen size achieved an increase in both the bulk and particle densities of the DDGS. The coefficient of internal friction was almost the same for both samples but cohesion was higher in Noramera samples (8.534 kPa). The DDGS obtained from Noramera Bioenergy Corp. contained dry matter (91.40%), crude fibre (4.98%), crude protein (37.41%), cellulose (10.75%), hemi-cellulose (21.04%), lignin (10.50%), starch (3.84%), fat (4.52%) and ash (5.16%); whereas the samples obtained from Terra Grain Fuels contained dry matter (87.69%), crude fibre (7.33%), crude protein (32.43%), cellulose (10.81%), hemi-cellulose (27.45%), lignin (4.37%), starch (4.18%), fat (6.37%) and ash (4.50%).

The combustion energy of the Noramera samples was 19.45 MJ/kg at a moisture content of 8.6% (w.b.) whereas the combustion energy of Terra Grain samples was 18.54 MJ/kg at 12.31% (w.b.) moisture content.

The durability of the pellets increased as the screen size decreased which is likely due to the fact that a smaller screen size produces more fine particles. This fill voids in the pellets and, hence, makes them more durable.

The length of the pellets produced from Noramera DDGS increased with a decrease in moisture content possibly because pellets formed at higher moisture content absorb less moisture. Therefore, the length does not increase as much. Lateral expansion occurred most with higher

temperature and lower moisture content and with lower temperature and higher moisture content. The length to diameter ratio of the pellets followed the same trend as the change in pellet length. The length of the pellets produced from Terra Grain also increased with a decrease in moisture content. The lateral expansion increased with increase in screen size and moisture content and also, with decrease in moisture content and increase in temperature. The length to diameter ratio increased with decrease in screen size and moisture content, similar to the change in pellet length.

The highest bulk density of Noramera pellets resulted from smaller screen size and lower moisture. The particle density increased with a decrease in screen size and an increase in moisture content. The highest bulk density of Terra Grain pellets occurred with an increase in temperature and decrease in moisture content. The highest particle density occurred with an increase in temperature and decrease in screen size.

The pellet hardness increased with a decrease in moisture content and screen size did not have any significant effect. The Terra Grain pellets were harder because they were subjected to steam conditioning. Steam conditioning helps to increase the hardness.

The pellet durability increased with a decrease in screen size and increase in moisture content. The steam conditioning also caused the higher durability in the Terra Grain pellets.

In terms of moisture absorption, the only significant factor was moisture content. Pellets with lower moisture content absorbed more moisture.

The ash content values of pellets were higher in Noramera samples than in Terra Grain samples because of high moisture content in Noramera samples. The combustion energy of the Noramera

pellets was higher than the Terra Grain pellets because of the high percentage of dry matter and lignin present in Noramera samples.

The emission results for both the sample pellets were similar. When the DDGS pellets were compared to wood pellets, emission of nitrous oxide was lower for wood whereas, carbon dioxide was higher.

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DEDICATION

This thesis is dedicated to my loving parents, my mother Supti Saha and my father Gobinda Saha who has raised me to be the person who I am today and was constantly supporting me throughout my studies. Also, this thesis is dedicated to my aunt Shefali Saha and my late uncle Narayan Saha who loved and cared for me unconditionally.

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

NOMENCLATURE

φ_i = angle of internal friction (degree)

ε = porosity (%)

ρ_b = initial bulk density (kg/m^3)

σ = normal stress (Pa)

ρ_t = particle density (kg/m^3)

μ = coefficient of friction

C_c= cohesion (kPa)

ADL = acid detergent lignin

ADF = acid detergent fibre

d.b. = dry basis (%)

df = degree of freedom

d_{gw} = geometric mean diameter or median size of particles by mass (mm)

d_i = nominal sieve aperture size of the i^{th} sieve (mm),

d_{i+1} = nominal sieve aperture size in next larger than i^{th} sieve (mm)

DDGS = distillers' dried grains with solubles

DDG = distillers' dried grains

e = correction for the heat of firing fuse wire (J)

H = energy content of the sample (J/kg).

H_g = energy content of standard benzoic acid (J/kg)

l/d = length-to-diameter ratio

m = mass of sample in combustion cap(kg)

m = mass of sample in the cylinder (kg)

m_a = mass of benzoic acid (g)

m_w = mass of water added to sample (g)

m_i = initial mass of sample (g)

m_i = mass on i^{th} sieve (g)

M_{wf} = final desired moisture content of sample (w.b.)

M_{wi} = initial moisture content of sample (w.b.)

MS = Mean square

n = number of replicates

n = number of sieves +1 (pan)

NDF = neutral detergent fibre

PDI = pellet durability of index

P_1 = pressure reading after pressurizing the reference volume (kPa)

P_2 = pressure reading after including volume of the cell (kPa)

R^2 = coefficient of determination

S.E. = standard error

SEE = standard error of estimate

S_{gw} = geometric standard deviation of particle diameter (μm)

S_{log} = geometric standard deviation of log-normal distribution by mass in ten-based logarithm, dimensionless

τ = shear stress (Pa)

V = volume of compacted sample at pressure P (cm^3)

V_0 = volume of sample at zero pressure (cm^3)

V_C = volume of cylinder (cm^3)

V_{cell} = volume of the cell (cm^3)

V_R = reference volume for the large cell (cm^3)

V_s = volume of solid (cm^3)

W = mass of sample (g)

W = energy equivalent of calorimeter (J/°C)

w.b. = wet basis (%)

W_d = moisture content (% d.b.)

W_i = mass on i^{th} sieve (g),

W_w = moisture content (% w.b.)

W_1 = mass of the ash (g)

W_2 = mass of oven-dry sample (g)

WDG = wet distillers' grains

1. INTRODUCTION

In recent years, there has been an increase in the cost of non-renewable fossil fuels. This has been a major issue for many energy-dependent countries, including Canada. There are two ways in which this issue could be solved, either reduce energy dependency or develop alternative methods of energy production. Biomass is a main source of energy, especially in rural areas where conventional sources of energy are not readily available. Biomass includes wood waste, agricultural waste, bagasse, industrial residue, alcohol fuels, sawdust, bio-solids, grass waste from food processing, crop waste, grasses, legumes, biological waste, etc. All of these could be used to produce biofuels (Rosentrater and Kongar 2009; Demirbas 2004). Currently, the material used most often in Western Canada is wheat. Producing fuel ethanol from wheat is very efficient and is relatively inexpensive compared to production from other biomass sources.

Distillers' dried grains with solubles (DDGS) are a co-product of the fermentation of wheat/corn that produces ethanol and carbon dioxide in biofuel and beverage ethanol industries. There is an annual fuel ethanol production of 502 million litres in Western Canada from seven fuel ethanol processing plants (Opoku et al. 2009). The production of ethanol in Western Canada has resulted in a high production of wheat DDGS. DDGS is a dry, granular bulk material, often with particle sizes less than 1.0 mm in diameter. Water activity values are often below 0.5, the thermal conductivity is typically near 0.7 W/m°C and the angle of repose can range from 25°-35° (Rosentrater 2006). Protein content of DDGS often varies from 26-34% (d.b.), fat ranges from 3-13% (d.b.), neutral detergent fibre (NDF) is between 25 and 50% (d.b.), and ash ranges from 2-10% (d.b.) (Rosentrater and Muthukumarappan 2006).

DDGS are typically dried to approximately 10% moisture content to ensure long shelf life and help transportation and are either sold as feed to livestock or shipped to other feed markets throughout Canada. With the increase in the cost of conventional fuels, the cost of transporting DDGS in granular form has increased since less material is being transported due to the low bulk density of DDGS. To reduce the cost of transportation, the bulk density of DDGS could be increased. Numerous kinds of biomass have been densified for use as a source of bio-fuel. Extrusion, pelletization and briquetting are the most common forms of densified biomass (Li and Lui 2000). The biomass can be made into pellets, cubes and briquettes by applying mechanical pressure, which helps the raw materials bind together and form pellets. Raw material properties such as particle size, bulk and particle densities, and chemical components have been studied during densification. In the densification process, grinding creates inter-particle bonding, as well as, well-defined shapes and sizes of pellets, briquettes, and cubes (Kaliyan and Morey 2006).

Pelleting is a manufacturing process commonly used to densify granular materials. It alters their particle size and shape and, as a result, product flowability and storage characteristics (Rosentrater and Kongar 2009). Biomass from DDGS can be converted into solid fuel so that it can be easily burned in furnaces to heat farmhouses. The pellets made from DDGS should meet quality criteria such as: high bulk density, high hardness, high durability, high gross energy, low moisture absorption, low ash content and low emissions.

The composition of DDGS, shelf life and transportability parameters are vital in terms of feed quality. Research has been performed regarding the nutritional and physical properties, and flowability of DDGS (Speihs et al. 2002; Ganesan et al. 2006). Components of DDGS can be used as value-added products in addition to feed.

Some examples include: removal of fibres from DDGS, biodiesel production from corn oil, biomass gasification, DDGS with zein protein in biocomposites, biomass densification as bio-fuels and cellulosic degradation of DDGS for further ethanol production (Singh et al. 2001b).

Biomass has a lower heating value than coal mainly due to the higher moisture content and in part due to the high oxygen content. Studies comparing biomass and coal burning have shown that cofiring biomass with coal, in comparison with single coal firing, helps reduce the total emissions per unit energy (Demirbas 2003). So, DDGS pellets could be used with coal or on their own as an alternative source of energy for burning in furnaces.

1.1 Objectives

The general objective of this research was to produce pellets from DDGS, a co-product of the ethanol industry using a pilot-scale mill. The specific objectives were:

1. to characterize the distillers' dried grains with solubles on the basis of physical and chemical properties with regard to particle size distribution, bulk and particle densities, heat of combustion, angle of internal friction and chemical composition;

2. to examine the characteristics of the pellets, i.e. particle size, bulk density, particle density, moisture absorption, hardness and durability, based on the parameters of screen size, moisture content and temperature.

3. to test the pellets for heat of combustion (gross energy), ash content, and fuel gas emissions.

1.2 Organization of the thesis

This thesis is organized in six chapters, specifically: Introduction, Literature Review, Materials and Methods, Results and Discussion, Conclusion and Recommendations for Future Work. In the Introduction chapter, general introduction about distillers' dried grains with solubles and their potential to be used as biofuels is discussed. The Literature Review chapter presents the agricultural grains used to produce ethanol around the world and the production and background of distillers' dried grains with solubles. The physical and chemical properties of various biomass grinds and DDGS, as well as the processing parameters affecting the compaction of the biomass grinds, are discussed. The physical properties of densified products and emission of biomass pellets are reviewed. Finally, the economics of pelleting are considered.

In the Materials and Methods chapter, the methods followed and the equipment used to measure the physical and chemical properties, and the heat of combustion and fuel gas emissions of the pellets are described. In the Results and Discussion chapter, the experimental results obtained for the physical and chemical composition of raw materials are presented and interpreted. Lastly, the Conclusion is followed by Recommendations for Future Work, which addresses issues that could be considered in future studies.

2. Literature Review

Distillers' dried grains with solubles (DDGS) are co-products of the dry-mill process in the ethanol industry. DDGS are studied for their physical and chemical properties to determine how these properties affect compaction of the DDGS. Compaction is a method in which loose raw materials are converted to a condensed mass by mechanical pressure. The commonly used methods are baling, cubing, briquetting and pelleting (Xu et al. 2008).

Pelleting of feed materials has been done for many years. Pelleted biomass has many advantages over granular biomass. The bulk density is increased. Flowability is improved because transformation of the physical form changes the angle of repose. Inter-particle friction is improved and consequently, less linking exists between particles. Pelleting of biomass lowers feed waste, dust generation and ingredient segregation (Rosentrater 2007). The major factors that affect pelleting include feed conditioning and ingredients, and pellet mill die geometry. However, one of the main concerns of pellet production is feed conditioning. Conditioning includes heating the feed particles, steam and moisture addition, and mixing. Appropriate conditioning prior to pelleting has numerous possible benefits, including increased pellet durability, and increased starch gelatinization and protein structures, which improve the binding of the materials. Steam addition aids in potential pasteurization by destroying salmonella, fungi, insect eggs, etc. There are also disadvantages to pelleting. There is an increased space requirement for the pelleting equipment; a steam source is required which increases electricity consumption; and finally, capital and operational costs are higher. However, the potential benefits outweigh the costs. These benefits can include improved feed conversion in livestock, and improved feed handling, storage and transportation (Rosentrater 2007).

2.1 Agricultural grains used for ethanol production worldwide

According to the OECD-FAO agricultural outlook report, (The Bioenergy Site 2010) the different agricultural grains/products used for global ethanol production (i.e., molasses, vegetable oil, coarse grains, sugar beet, wheat and sugar cane) is expected to increase intensely from 2007 to 2019. The advancement of ethanol production over the projection period indicates that the major feedstock for ethanol production will continue to be from coarse grains. Nearly 40% of the increase in worldwide ethanol production may be due to an increase in the production of ethanol based on sugar cane, mainly from Brazil. Biomass based second generation ethanol is only expected to develop in the latter years of the projection period. Roots, tubers and molasses could be used as feedstocks for ethanol production in developing countries. Wheat, coarse grains and sugar beet may be used in the European Union to produce ethanol (The Bioenergy Site 2010). Approximately 93,000 million litres of ethanol was produced worldwide in 2010 and the world price for ethanol is 47.44 USD/hL (OECD-FAO Database 2010). Corn is the chief grain used in wet mills and dry-grind ethanol plants as it has high fermentable starch content compared to other feedstocks. However, some ethanol plants use sorghum, or blend corn with barley, wheat, or sorghum to make ethanol and distiller's grains with solubles. This depends on geographical location, cost, and availability of these other grains relative to corn (Shurson and Noll, 2005). In the United States, 5 to 8 million metric tonnes of barley is produced and if 30 to 50% of available barley could be used for ethanol production, then approximately 0.5 billion gallons of ethanol could be produced (Hicks et al. 2006).

2.2 Agricultural grains used for ethanol production in Canada

Wheat and corn are the main agricultural grains being used for ethanol production in Canada. Canada produces 22 to 24 million tonnes of wheat per year with the majority coming from Saskatchewan. 14 to 17 million tonnes of wheat crops are exported, 2.6 to 2.9 million tonnes are used for food domestically, and 3.6 million tonnes used for feed and dockage with a carry over of 4 to 5 million tonnes. Canadian wheat-based ethanol is produced in Western Canada. The dry milling process of ethanol production produces nearly 365 litres of ethanol, 290 kg of DDGS and 290 kg of carbon dioxide from each tonne of wheat. The price of DDGS is 150-200 USD/tonne. Large ethanol plants have a capacity of 100 million litres whereas small plants can produce 20 to 25 million litres. As higher yielding wheat varieties are produced, it is estimated that about 4 million tonnes of wheat could be used for ethanol production without risking other feed or food supplies and production. This amount of wheat will produce 1.46 billion litres of ethanol and 1.16 million tonnes of DDGS, yearly. Ethanol plants using wheat as the feed stock are Permolex, Red Deer; Husky Energy, Lloydminster; Terra Grain Fuels, Belle Plaine; Pound Maker, Lanigan; Noramera Bioenergy, Weyburn; Northwest Bio-energy, Unity; and Husky Energy, Minnedosa (Canadian Renewable Fuel Association, Government of Alberta 2010).

The corn-based ethanol industry is based in Central Canada. On average, a dry mill plant produces 10.7 litres of ethanol and 17.5 lbs. of DDGS from one bushel of corn. Overall, 40 million tonnes of DDGS are produced from 5 billion bushels of corn per year. Greenfield Ethanol (Hensall, Tiverton, Johnstown, Vareness), Collingwood Ethanol, Collingwood and Suncor Energy, St. Clair plants uses corn as the feedstock for ethanol production. The combined capacity of these ethanol plants is approximately 1450 million litres of ethanol (Canadian Renewable Fuels Association 2010).

Approximately 1575 million litres of ethanol was produced in entire Canada and the producer price of ethanol is 56.69 USD/tonne (OECD-FAO Database 2010). Barley production in Canada is about 14 million metric tonnes. If 30 to 50% of the available barley could be used for ethanol production then an additional 0.8 gallons of ethanol could be produced (Hicks et al. 2006).

2.3 Agricultural grains used for ethanol production in Saskatchewan

Wheat is the most common crop in Saskatchewan. Therefore, most of the ethanol plants use wheat as feedstock. Husky Energy, Lloydminster and Noramera Bioenergy, Weyburn use both wheat and corn as their feedstock for ethanol production. However, Northwest Bio-Energy, Unity, Pound-Maker Agventures, Lanigan and Terra Grain Fuels, Belle Plain, use only wheat. In Saskatchewan, three existing ethanol plants produce close to 345 million litres of ethanol per year and the amount of wheat DDGS available for commercial sale is approximately 300,000 metric tonnes (Canadian Renewable Fuels Association 2010).

2.4 Background of DDGS

Distillers' dried grains with solubles are a product of the dry-mill ethanol plants in contrast to the wet milling process, which produces gluten feed and meal. Numerous methods are involved in ethanol production including grinding, cooking, liquefaction, fermentation and distillation. After the ethanol has been distilled off, the residual material is known as whole stillage.

Whole stillage is centrifuged into two fractions: coarse solids, or wet distillers' grains (WDG); and thin stillage. WDG can be fed to livestock in the wet form, or dried to produce dried distiller's grains (DDG). Thin stillage, can be used or evaporated to produce condensed distillers'

solubles, or syrup. This syrup is then added to the dried distillers' grains to produce distillers' dried grains with solubles (DDGS) (Christiansen 2009).

2.4.1 Production of DDGS

This section will outline the detailed dry-milling process of ethanol production from wheat. "First, water is added to the ground grain to form a slurry and it is then heated to a moderately high temperature in the liquefaction process. Heat stable starch degrading enzymes (i.e., alpha-amylase) are also added during this step. After liquefaction, the mash is cooled and gluco-amylase is added to convert the liquefied starch to fermentable sugars, producing ethanol and carbon dioxide. After fermentation occurs, the beer from the fermentation tanks is directed through a distillation system, which removes the ethanol, leaving a product known as 'whole stillage'. The whole stillage is centrifuged into wet distillers' grains and thin stillage. An evaporation step removes excess water from the thin stillage, leaving condensed syrup. This is then mixed with the dried distillers' grains to produce dried distillers' grains with solubles" (Wheat DDGS 2010).

The shelf life of wet distillers' grains with solubles is less than one week as their moisture content is around 65-70%. So the market areas of these products are within limits of 98 kms of dry-mill ethanol plant. Whereas, the market for distillers' dried grains with solubles (DDGS) is worldwide. Ethanol plants, on average, sell two-thirds of their DDGS but the shelf life to store the DDGS is only two weeks. Therefore, producers depend upon reliable transportation. The market for truck transportation is 402 kms from the plant on an average. Rail is used to ship DDGS longer distances, which helps in expanding the domestic market. In the United States,

only 15% of DDGS are exported, mainly to the European Union, Mexico, Central and South America, the Caribbean, and Southeast Asia (Christiansen 2009).

2.4.2 Current use of DDGS

The majority of DDGS are sold as a feed ingredient for livestock and poultry feeds. It can be used as feed for dairy and beef cattle along with swine (Shurson and Noll, 2005). By varying the nutritional values of the DDGS, it may be used as feed material for other livestock. DDGS have also been used as feed materials in aquaculture as an alternative protein source for fishes (The Fish Site 2010).

2.4.3 Alternative use of DDGS

DDGS, as a biofuel, can be used as an alternate energy source. DDGS can be compacted into pellets and used in place of commercial wood pellets for burning. Due to the large quantities of DDGS produced every year, they are readily available in the market. It has been estimated that about 4 million tonnes of wheat could be used for ethanol production without risking other feed or food supplies and production. This amount of wheat will produce 1.46 billion litres of ethanol and 1.16 million tonnes of DDGS (Canadian Renewable Fuels Association 2010).

Pellets can be produced in a pellet mill. China is the major producer of pellet mills in the world. Two basic pellet mills manufactured in China are the flat die biomass energy wood pellet mill, which has a capacity of 500 kg/h, and the ring die biomass energy wood pellet mill with a capacity of 200 kg to 3 tonne/h. Some of the pellet manufacturers in Canada are Lawson mills biomass solution; PE and IMG pellet system, Burnaby, BC. Pellets produced are approximately

$10/tonne. The price of a pellet mill for domestic purpose is between 1000-4000 USD with a capacity of 150 to 200 kg/h.

If DDGS were pelletized before shipping, the increased bulk density of the pelletized DDGS would result in transportation cost savings and would improve DDGS flowability issues. Ag Fuel & Feed, a company in the United States can produce a pellet with a bulk density of 650.35 kg/m^3, 28 percent more dense than un-pelletized DDGS, and the pellets flow better than unpelletized DDGS. Pelletizing DDGS alleviates handling problems. This might help to open export markets where customers are having difficulty unloading and transporting bulk products. The pellets flow better in screw conveyers and bucket elevators and would be easier to unload from railcars (Christiansen 2009).

2.5 Biomasses used for making pellets and their characterizations

Various biomass materials have been used to make pellets, cubes or briquettes for many years. Biomass, including wheat straw, corn stover, peat moss, oat hulls, flax shives, wood chips, barley straw, crop waste, grasses etc., has been used for densification (Tabil 1996; Tabil and Sokhansanj 1996; Mani et al. 2004a; Rhen et al. 2005; Mani et al. 2006b; Mozammel et al. 2006; Kaliyan and Morey 2006; Shaw and Tabil 2006). Raw material properties such as particle size reduction, particle density, bulk density, angle of internal friction and moisture content affect the densification and properties of the products.

2.5.1 Physical properties of various biomasses

Size reduction of biomass is an important pre-treatment for the densification process and energy conversion. The geometric mean diameter and particle size distribution of biomass grinds affect

densification and binding characteristics and are useful information for the design of pneumatic conveyors and cyclones (Mani et al 2002). Wheat and barley straw grinds have a geometric standard mean diameter of 0.64 and 0.69 mm, respectively when ground using a hammer mill with a screen size of 3.2 mm (Mani et al 2006b). Particles distributed in this range were found to be suitable for pelleting (Mani et al. 2004a). Mani et al. (2006b) also observed that there was an increase in bulk density from 97 to 121 kg/m^3 and particle density from 1030 to 1340 kg/m^3 when the geometric mean particle diameter was reduced from 0.64 to 0.28 mm in a wheat straw grind. Decreasing the geometric particle size of corn stover from 0.80 to 0.66 mm resulted in about 5 to 10% increase in the relaxed density of briquettes and increased the briquette durability from 50 to 58% at 100 MPa pressure, and from 62 to 75% at 150 MPa pressure (Mani et al. 2006b).

The bulk and particle density of wheat straw with a hammer mill of screen size 3.175 mm was 112.56 kg/m^3 and 1286.06 kg/m^3, respectively, whereas the bulk and particle density of flax shives with a hammer mill of screen size 6.35 mm was 107.99 kg/m^3 and 1346.14 kg/m^3 (Shaw and Tabil 2006). The bulk densities of small size wood chips varied from 180 to 314 kg/m^3 whereas of large size wood chips from 158 to 308 kg/m^3 respectively, with moisture content ranging from 10.7 to 55.71% wet basis (w.b.) (Mozammel et al. 2006). Mozammel et al. (2006) also reported that the bulk density of barley straw at a moisture content of 8.45% varied from 24 to 54 kg/m^3 depending on the particle size. Shaw (2008) studied the bulk and particle densities of poplar, pre-treated poplar, and wheat straw at two moisture levels (i.e., 9 and 15% w.b.) using screen sizes of 0.8 and 3.2 mm. Both the bulk and particle densities of the feedstock decreased with an increase in moisture content and screen size (Shaw 2008).

Corn stover grind with a low moisture content of 7% had higher compressibility than a grind with a high moisture content of 15%. At a high moisture content, corn stover grind exhibited high resistance to compression, perhaps due to the incompressibility of water (Mani et al. 2004b). This shows that biomass with low moisture content is more suitable for pelleting and briquetting. It was also reported that at 12% moisture content corn stover produced higher density pellets of 1136 kg/m^3 (Mani et al. 2006b). Corn stover produced highly durable (90%) and stable briquettes at 5 to 10 % moisture content and 15 MPa applied pressure. The durability decreased at 15% moisture content at all applied pressures because more surface cracks and axial expansion were observed on the briquettes (Mani et al. 2006a). Below 12% (w.b.) moisture content, stable wafers could not be formed. With increasing moisture content, durability and density increased to a maximum at about 14% (w.b.) moisture content and then decreased (Orth and Löwe 1977). Wheat straw briquettes showed high durability at 15% moisture content (Smith et al. 1977). Coates (1999) studied cotton briquettes and showed that more durable and stable briquettes were formed with 10-15% moisture content. For high quality alfalfa pellets, a moisture content of 8 to 9 % (w.b.) is recommended (Tabil 1996). Generally, moisture contents of 11 to 12 % (w.b.) are used for wheat and corn-based feed pelleting (Adapa et al. 2009).

The chemical composition of various biomass materials is given in Table 2.1. Mani et al. (2006b) performed the proximate analysis of wheat straw, barley straw, corn stover, and switchgrass. Canola and oat straw was studied by Adapa et al. 2009. The adhesive properties of thermally softened lignin are considered to be the strength characteristic of pellets and briquettes made of lignocellulosic materials (Shaw and Tabil 2007).

Table 2.1 Chemical compositions of various biomass materials

Biomass	Crude Protein (% d.b.)	Crude Fat (% d.b.)	Lignin (% d.b.)	Cellulose (% d.b.)	Hemicellulose (% d.b.)	Ash (% d.b.)
Wheat straw	5.70	1.61	7.61	42.51	22.96	8.32
Barley straw	6.60	1.33	6.81	42.42	27.81	10.72
Corn stover	8.70	1.33	3.12	31.32	21.08	7.46
Switch grass	1.59	1.87	7.43	44.34	30.00	5.49
Canola straw	6.53	0.69	14.15	42.39	16.41	2.10
Oat straw	5.34	1.65	12.85	37.60	23.34	2.19

Frictional characteristics are very important in properly designing agricultural product handling equipment for solid flow and structures for storage of the materials (Mohsenin 1986). Chung and Verma (1989) reported that the static and dynamic coefficients of friction, in general, increase with an increase in the moisture content of the grain. , Shelled corn tends to have a higher kinetic coefficient of friction than soybeans and rubber exhibits the highest coefficient, followed by plywood and galvanized sheet metal surfaces (Tsang-Mui-Chung et al. 1984). Chung and Verma (1989) found that the moisture content of the samples was more influential on the static coefficient of friction than the surface materials tested. However, surface materials were more influential on the dynamic coefficient of friction than the moisture content. The moisture content was more effective for improving friction coefficients for unshelled peanuts than for soybeans and kidney beans.

The coefficient of friction for wheat on steel was found to vary with moisture content, overburden pressure and sliding velocity (Thompson and Ross 1983). The coefficient of friction increased with an increase in moisture content from 8 to 20% but decreased at a moisture content of 24%. As the overburden pressure increased from 7 to 172 kPa, the coefficient of friction decreased but it increased with an increase in sliding velocity from 0.06 to 6 m/h (Thompson and Ross 1983). Thompson and Ross (1983) also reported that an increase in relative humidity and an increase in grain-surface moisture content increase the coefficient of kinetic friction of wheat on metal surfaces. The coefficient of kinetic friction for wheat on a metal surface changes quickly under nonequilibrium moisture content test conditions, most likely due to wetting or drying of the kernel surface (Snyder et al. 1967). On smooth galvanized steel, the friction decreased as the number of tests increased. On smooth steel and corrugated steel, the friction decreased with an increase in normal pressure (Molenda et al. 2000).

2.5.2 Machine variables used for compacting

A basic method for reducing the volume of forage and granular-farinaceous fodders is by pelleting. The pressure required for pelleting in commercial pellet mills varies between 500 and 1500 bar (50 to 150 MPa) (Sitkei 1986). However, the ideal pressure required to form satisfactory pellets, wafers or briquettes depends on the properties of the raw materials.

Pressures between 31.08 and 136.77 MPa were considered for determining the density of biomass pellets made from wheat and barley straws, corn stover, and switchgrass using a single pelleter (Mani et al. 2004c). For all materials, except the corn stover, an increase in the compressive force resulted in an increase in the compacted density of the pellets using various

screen sizes (i.e., 0.8, 1.6, and 3.2 mm). For corn stover grinds, there was no significant effect on the pellet density.

Mani et al. 2004c also reported that the maximum compact density was attained easily for corn stover and less pressure was required for densification. Oak sawdust and pine sawdust pellets were formed at a range of 34 to 138 MPa pressure with durability in the range of 71.2 to 98.3% (Kaliyan and Morey 2008).

Preheating temperature is a very important factor in the production of pellets, since it affects durability, hardness and moisture absorption. Rhen et al. (2005) used a wide range of temperature from 26 to 144 °C on Norway spruce pellets. It was concluded that high temperature and low moisture content were the most important variables for increasing the compression strength and dry density of the pellets. Shaw and Tabil (2007) studied flax shives, oat hulls and wheat straw pellets and found that the pellet densities increased with an increase in die temperature from 60 to 100°C. Kaliyan and Morey (2008) varied the preheating temperature from 65 to 100°C for studying various biomass products. This aided in manufacturing high quality products by pelleting, briquetting or cubing. In alfalfa pellets, Tabil and Sokhansanj (1996) found that the pellet temperature significantly affected the durability. It was necessary to condition the grinds at 90°C or higher to ensure that the pellet temperature would also be high. This promoted better bonding of the particles during pelting and, therefore, produced high quality pellets with high durability.

Steam conditioning the biomass before pelleting is important. Using steam to add heat and moisture to the biomass grind increases pellet production rate and improves pellet quality, durability, and hardness. Pellet durability increased to 96.5% due to steam conditioning and

fines were reduced during handling, transportation and feeding (Skoch et al. 1981). Tabil (1996) found that steam conditioning of alfalfa grinds at temperatures of 92°C and higher resulted in better quality pellets.

It was also found that increasing the conditioning temperature resulted in an increase in durability and a decrease in energy consumption for the pelleting process (Tabil 1996). Hill and Pulkinen (1988) reported that using steam conditioning to raise the temperature of alfalfa mash from 60 to 104°C resulted in a 30 to 35% increase in alfalfa pellet durability. In sun-cured alfalfa at a grind size of 3.20 mm, Adapa et al. (2004) used steam conditioning to form pellets with an inlet steam temperature was 118°C. In alfalfa grinds, the moisture content of 8.5 to 10% (w.b.) was suitable for making pellets (Tabil 1996). Commercial pelleting plants maintain the moisture content within a range of 12 to 16%, usually by adding steam. In hay, Dobie (1959) observed that for a moisture content of 10% (w.b.), the operation of the machine was critical and capacity was reduced, as the pellets formed were too dense and hard. Also, moisture above 18% (w.b.) is impractical for pelleting hay because fine grinding becomes difficult.

Steam conditioning time also affects the physical quality of the densified feedstocks and the energy consumption of the pellet mill. Tabil (1996) reported that a steam conditioning time of 17 to 20 seconds was sufficient to achieve the desired level of moisture and temperature for processing alfalfa pellets. In pelleting animal feeds, increasing the residence time of the mash in the steam conditioning chamber above 30 seconds slightly reduced pellet durability (Vest 1993).

Die geometry and die speed greatly affect the pellet density, hardness, durability and the specific energy required to form pellets. Heffner and Pfost (1973) studied the effect of die geometry on pellet durability. They concluded that the die size, pellet durability and gelatinization are related.

Pellets produced on the smallest die having length to diameter (l/d) ratio of 9.33 had the highest durability followed by the medium sized die (l/d=9) and the largest die (l/d=8). Smaller dies produced more gelatinization, more durable pellets and increased the nutritive value of the pellets.

Hill and Pulkinen (1988) found similar results. More durable pellets were formed using small dies (6.4mm) and less durable pellets were formed using large dies (12.7 mm). Length-to-diameter (l/d) ratios of 4.1 and 7.31 produced pellets with mean durabilities of 49.8 and 65.8%, respectively. Higher l/d ratios resulted in more durable pellets (Tabil and Sokhansanj 1996). An increase in alfalfa pellet durability is a function of the increased pressure and frictional heating of the ground particles in the die during the pelleting process (Tabil 1996).

Die speed also influences pellet densification and the quality of pellets produced using a pellet mill. Leaver (1985) stated that die speeds between 250 and 300 rpm are suitable for the production of small diameter pellets. Tabil (1996) produced alfalfa pellets at two die speeds (250 and 316 rpm), with die diameter of 6.1 mm. It was observed that there is an increase in the durability of the pellets created at the lower die speed. Pelleting using high die speeds (501 and 565 rpm) requires higher specific energy for the pellet mill motor to complete densification and it was determined that the high rotational speeds created more centrifugal force which affected the ability of the particles to flow through the die (Tabil 1996).

2.5.3 Quality property of the pellets

Previous studies regarding dimensional stability, density, hardness, durability and moisture absorption of pellets are reviewed in this section. A change in the dimensional stability of pellets (i.e., change in length and diameter) is affected by water absorption and the breakage of bonds in

the particles formed during the compaction process. Shaw (2008) considered pellets produced from raw poplar and wheat straw and found that they expanded in both diametrical and longitudinal directions. However, pellet expansion decreased in both axes with an increase in the die temperature and a decrease in the feedstock moisture content and particle size (Shaw 2008). Shaw and Tabil (2007) studied flax shives, wheat straw and oat hull grinds. They reported that diametrical pellet expansion was reduced by an increase in die temperature and biomass moisture content. Moisture content and particle size did not have a considerable effect on the diametrical expansion of flax shive pellets. In general, the pellets expanded significantly more in the longitudinal axis than in the diametrical direction. The longitudinal expansion decreased with an increase in the die temperature and a decrease in biomass moisture content. There was not a significant effect on the longitudinal expansion of oat hull pellets due to moisture content. Decreasing the particle size reduced the longitudinal expansion of oat hull pellets; whereas it did not have an effect on flax shive and wheat straw pellets (Shaw and Tabil 2007). Colley et al. (2006) found that there was an increase in the diameter of switchgrass pellets by 8% and a decrease in the length by 17% as the moisture content was increased. Bulk and particle densities also decreased by 24% and 16%, respectively, as the moisture content of the pellets increased.

When the hardness of switch grass pellets was considered, Colley et al. (2006) found that it decreased from 30.21 to 21.6 N with an increase in moisture content. Moisture disrupts particulate bonds, which leaves the pellets weak and susceptible to breakage (Colley et al. 2006). In alfalfa pellets, it was found that the pellets produced without binders from intermediate and high quality chops had a higher hardness compared to pellets from low quality alfalfa (Tabil 1996).

Durability of the material products is a very important property as it helps in determining resistance to impact forces and vibrations during transportation, storage and handling. Many devices exist for evaluating the durability of pellets. Thomas and van der Poel (1996) discussed different instruments including the Pfost tumbling can, Holmen tester, and sieving device.

The tumbling can method can be used with variations in the speed of tumbling, length of tumbling time, sieve size and the amount of samples tumbled (Richards 1990; Raghavan and Conkle 1991). For determining the durability of very hard pellets such as dairy feed pellets, the tumbling can method can be modified by adding steel nuts, bolts, or ball bearings along with the pellets before tumbling (Winowiski 1998). Mani et al. (2006a) measured the durability of corn stover using a DURAL tester and found that corn stover produced highly durable (90%) and stable briquettes at moisture contents of 5 to 10 % and 15 MPa applied pressure. However, the durability decreased at moisture content of 15% for all values of applied pressure. More surface cracks and axial expansion were observed on the briquettes. Switch grass briquettes made at room temperature had zero durability but ones produced at elevated temperatures of 75 to 150°C and pressure of 150 MPa exhibited relaxed densities of 834 to 1065 kg/m^3 and durability between 55 and 67% (Kaliyan and Morey 2006). In another test of switch grass, maximum durability of 95.91% was obtained when the pellets had a moisture content of 8.6% (w.b.) (Colley et al. 2006). Coates (1999) studied cotton briquettes and showed that more durable and stable briquettes were formed with 10-15% moisture content. Oak sawdust and pine sawdust pellets were formed at a range of 34 to 138 MPa pressure with durability in the range of 71.2 to 98.3% (Kaliyan and Morey 2008). Skoch et al. (1981) found that pellet durability increased to 96.5% due to steam conditioning. It was also reported that by steam conditioning and raising the temperature of alfalfa mash from 60 to 104°C a 30 to 35% increase in alfalfa pellet durability

occurred (Hill and Pulkinen 1988). The durability of alfalfa pellets at three different qualities of chops were tested using both Dural and Stein breakage testers and it was found that pellets made from high quality chops were the most durable (Tabil 1996).

The durability of Norway spruce pellets increased with an increase in temperature and a decrease in moisture content. The durability was determined as compression strength of a single pellet and measured with a compression tester (Rhen et al. 2005).

An increase in air temperature and relative humidity will cause an increase in the moisture absorption of pellets during storage, transportation and handling. In a study of Norway spruce pellets, Rhen et al. (2005) found that the highest moisture uptakes occurred in the samples with the lowest initial moisture content at compression and vice versa. They also found that the minimum moisture absorption occurred at a temperature of about 90°C. It was concluded that the initial moisture content of the raw material is the most important variable for controlling moisture uptake and a conditioning period of 25 h is adequate for equilibration of moisture content in a pellet. Colley et al. (2006) found that relative humidity has a significant effect on the moisture uptake and final moisture content in switchgrass pellets. Tabil (1996) performed water absorption studies on alfalfa pellets based on the quality of the chops. Pellets produced from intermediate quality chops took longer to absorb moisture than the pellets from other quality chops.

In section 2.5, various biomass materials were reviewed on the basis of physical properties, machine variables used for making pellets and the quality properties of the pellets. Since the raw material being used in this study is dried distillers' grains with solubles (DDGS), physical and

mechanical properties; the machine variables used for making DDGS pellets and quality properties of DDGS pellets are reviewed more thoroughly in the following section.

2.6 Physical properties of DDGS

The physical properties of DDGS to be discussed are: moisture content, density and proximate analysis. The raw material properties are important as they help in determining the steps required in the processing of the materials.

2.6.1 Moisture content

Moisture content is the amount of water (in any form) in any material or substance. Moisture content can affect the specific energy required to compact biomass materials as well as properties such as densities, angle of friction, specific heat capacity, force-deformation characteristics, and thermal conductivity. The initial moisture content of biomass grinds also has a significant effect on the densification process and final product properties (Rentsen 2010). The moisture in biomass both acts as a facilitator of natural binding agents and a lubricant (Adapa et al. 2009). Optimal moisture content of the biomass is essential for producing stable and durable pellets.

In corn-based DDGS, researchers have shown that an increase in moisture content from 15 to 25 % (w.b.) results in a 28.2% increase in durability and an 8.3 and 8.5% decrease in specific gravity and porosity, respectively (Chevanan et al. 2008). Rosentrater (2006) studied corn DDGS samples that had moisture contents ranging from 13.2 to 21.2% (d.b.) but a maximum moisture content of approximately 12% (d.b.) is generally recommended for feed products as transportation costs are minimized and microbial growth is prevented. In wheat DDGS pellets, moisture contents ranging from 11 to 16% (w.b.) were considered and the durability ranged from

60% to 93% with bulk densities ranging from 436 to 529 kg/m^3 (Opoku et al. 2009). Another test on wheat based DDGS with moisture content ranging from 5.10 to 11.80% (w.b) had a durability index ranging from 91.4 to 99.9 % with pellet density in the range of 860 to 1080 kg/m^3 (Tumuluru et al. 2010).

2.6.2 Bulk Density

Bulk density is the ratio of the mass of a collection of discrete pieces of solid material to the sum of the volumes of: the solids in each piece, the voids within the pieces, and the voids among the pieces of the particular collection (Webb 2001), whereas the particle density is the mass of the particle divided by its volume excluding open and closed pores (BSI 1999). Bulk density is a crucial factor in the design and operation of loading vessels, such as bins, tanks, trucks and rail cars (Rosentrater 2006)

The bulk densities of corn DDGS were found to be in the range of 467.7 to 509.38 kg/m^3 when the moisture content ranged from 3.54% (d.b.) to 8.21% (d.b.) (Bhadra et al. 2007). Rosentrater (2006) found bulk densities of corn DDGS varied from 389.3 to 501.5 kg/m^3 when the moisture content ranged from 13.2 to 21.2% (d.b.). The bulk densities of wheat DDGS tested on a range of moisture content from 8.56% (w.b.) to 10.46 % (w.b.) were found to be in the range of 360.1 to 364.4 kg/m^3 (Opoku et al. 2009).

2.6.3 Proximate Analysis

The basic constituents of biomass are starch, protein, sugar and non-starch polysaccharides, fat, fibre and inorganic matter. Processing conditions during commercial pelleting (i.e., temperature, pressure, steam, etc.) cause changes in individual constituents to positively or negatively affect

the pellet quality. Natural binding agents, including starch, protein and lignin, are considered to improve pellet quality (Shaw and Tabil 2007). Starch has been reported to act as a binding or adhesive agent and provide a lubricating effect during the pelleting process. Higher protein content increases the pelleted feed durability whereas oils produced lower the quality of the pellets. Lignin is the component that permits adhesion in the wood structure. It has been reported to exhibit thermosetting properties upon melting. The adhesive properties of thermally softened lignin are considered to improve the strength of densified products made of lignocellulosic materials (Shaw and Tabil 2007).

Some of the chemical compositions of grains and their DDGS are given in Table 2.2. Rasco et al. (1987) studied soft white wheat, hard red wheat, and corn and their DDGS. Sorghum and sorghum DDGS was studied by Wu and Sexson (1984).

Table 2.2 Chemical compositions of various grains and their DDGS

Biomass	Protein (% d.b.)	Fat (% d.b.)	Fibre (% d.b.)	Ash (% d.b.)	Starch (% d.b.)
Soft white wheat	6.8	1.7	2.9	1.5	87.1
Soft white wheat DDGS	19.6	3.8	7.6	8.4	60.7
Hard red wheat	14.1	1.8	2.6	1.8	79.7
Hard red wheat DDGS	33.9	2.5	6.8	6.8	49.9
Corn	7.4	3.7	2.1	1.3	85.5
Corn DDGS	23.0	9.0	6.3	10.1	51.5
Sorghum	10.9	3.2	2.2	1.5	73.9
Sorghum DDGS	45.3	12.3	11.6	2.1	5.7

The protein content of corn-based DDGS often varies between 26 and 34% (d.b.), fat ranges from 3 to 13% (d.b.), neutral detergent fibre (NDF) is between 25 and 50% (d.b.) and ash ranges from 2 to 10% (d.b.) (Rosentrater and Muthukumarappan 2006). Another study on corn-based DDGS found a composition of 29.93% crude protein, 10.5% crude fat, 11.07% total starch, 10.22% crude fat (CF), 36.74% NDF, 16.2% acid detergent fibre (ADF) and 12.82% crude ash (Bhadra et. al, 2007). A comparative study of wheat DDGS, corn DDGS and a wheat/corn blend DDGS (i.e., wheat: corn=70:30) were studied by Nuez-Ortín and Yu (2009). It showed that ash content was highest in wheat DDGS (5.12%) and lowest in corn DDGS (4.32%). Crude fat was highest in corn DDGS (16.53%) and lowest in wheat DDGS (4.98%). The NDF value was highest in the blend DDGS (51.50%) and lowest in wheat DDGS (48.07%). The ADF value was highest in corn DDGS (14.68%) and lowest in the blend DDGS (10.80%). Lignin was found to be higher in wheat DDGS (4.32%) and lower in corn DDGS (2.80%). Hemicellulose was higher in the blend DDGS (40.70%) and cellulose was higher in corn DDGS (11.88%). Total starch and crude protein both were found to be highest in wheat DDGS with values of 6.32% and 39.32%, respectively. These results show that wheat DDGS are a better component for biomass pelleting as they have higher values of starch, protein and lignin. The wheat DDGS studied by Opoku et al. (2009) for pelleting had a composition of dry matter (92.08%), crude protein (39.30%), NDF (51.53%) and ADF (20.34%). The composition of feed materials differs in terms of protein, fibre, fat and starch and these changes influence the ability to produce pellets (Rosentrater 2007).

2.7 Mechanical properties of DDGS

Mechanical properties of DDGS, i.e., the particle size reduction of the raw material and the angle of repose of the materials are reviewed in this section.

2.7.1 Size reduction

Particle size reduction increases the total surface area of the material and the number of contact points for inter-particle bonding in the compaction process. Hammer mills are generally used for size reduction of the materials, as they are relatively cheap, easy to operate and produce the wide size range of particles that is required for the densification of ground materials. The performance of a hammer mill is measured by the energy consumption and the geometric mean diameter and particle size distribution of the ground product (Mani et al. 2002). The parameters used in particle size analysis of DDGS are maximum diameter, minimum diameter, average area and the roundness values. Roundness is the degree of abrasion of a grain particle as shown by the sharpness of its edges and corners. Bhadra et al. 2007 sampled the DDGS from screen sizes ranging from 149μm to 2.38 mm. The mean geometric diameter was 0.91 mm, average area was 9.15 mm^2 and average roundness was found to be 50.73% (Bhadra et al. 2007). Xu et al. (2008) used DDGS particle sizes ranging from 0.2 to 2.36 mm for pelleting. Rosentrater (2008) used DDGS with geometric mean diameters between 0.65 and 0.93 mm. Generally, the finer the particles, the better the resulting pellets, since moisture and heat have the ability to penetrate more thoroughly during conditioning. Rosentrater (2007) recommended that the optimal particle size be between 500 and 600 μm.

2.7.2 Angle of repose

The coefficient of friction between granular materials is equal to the tangent of the angle of internal friction for that material and the angle of repose is the angle with the horizontal at which the material will stand when piled (Mohsenin 1986). The internal angle of friction can sometimes be approximated by the angle of repose of the material and is reasonable only when the cohesive

strength of the material is believed to be negligible (Seville et al. 1997). The angle of repose for rice on rice was studied and it was found that the angle increased very rapidly when moisture content increased to 16 or 17% (Mohsenin 1986). Mohsenin (1986) also studied the effect of the moisture content of wheat on the angles of repose and internal friction. They found that when the moisture content increased to 19.3%, there was a corresponding increase in the angle of repose to 41°. Rosentrater (2008) found the angle of repose in DDGS to range from 13.10° to 20.06°. In a different study by Bhadra et al. (2007), the angle of repose was found to range from 25.7° to 47.04°. The angle of repose gives an idea of grain structure, i.e., the higher the value, the lower the flow rate (Bhadra et al. 2007).

2.8 Processing aspects of DDGS pellets

After the raw materials are tested for their physical properties, pelletization is performed. Pelleted feed offers several advantages over granular feed materials; including increased bulk density and flowability because the altered physical form changes the angle of repose and inter-particle friction, and produces less bridging between particles.

2.8.1 Equipment used to make DDGS pellets

Pellet mills are used to produce DDGS pellets commercially. A California pellet mill, consisting of a die and roller assembly, is generally used for making DDGS pellets. Wood pellet mills could also be used to make DDGS pellets. Single pelleting equipment, which consists of a die and plunger assembly with stainless steel base along with a heating element, is also used.

2.8.2 Processing parameters for DDGS pellets

Biomass densification (pelleting) factors such as pressure, temperature, steam conditioning, die size and die speed affect the performance of the densification equipment and the quality of the final products. These factors also have a substantial influence on the pellet properties during and after pelletization.

For pelleting of DDGS, Xu et al. (2008) studied temperatures in the range of 100° to120°C with die pressures ranging from 12.5 to 37.5 MPa and dwell time ranging from 5 to 15 sec. The durability of the pellets increased as the pressure increased from 12.5 to 37.5 MPa. This could have occurred because under high pressure, the natural binding agents like starch, protein and lignin in the DDGS were squeezed out of the particles, resulting in strong interparticle bonding (Kaliyan and Morey 2008). The optimal temperature and pressure for forming pellets were found to be 107°C and 36.8 MPa, respectively, and there was no significant effect due to dwell time (Xu et. al 2008).

Pre-heating temperature affects the quality of the pellets. When the optimum temperature is not reached, the pellets formed are very crumbly in nature. Preheating temperature is limited to 300°C to avoid decomposition of biomass materials (Kaliyan and Morey 2008). In wheat DDGS, a temperature between 50°C and 80°C and low moisture content of about 5.1% have resulted in the maximization of pellet density and durability, and minimization of pellet moisture content (Tumuluru et al. 2010).

Steam conditioning helps in activation of natural and artificial binders (where used), gelatinization of starch and release of natural lubricants. It also increases moisture content and

improves feeding conversions, which, in turn, softens the fibre in the feedstock (Robinson 1984). Steam conditioning also helps to reduce germ and bacterial counts (Kaliyan and Morey 2008).

Steam conditioning may include both vapor diffusion and condensation. Vapor diffusion from the pressurized steam causes inter-particular voids in the grind, while condensation of vapor on the surface of the mash changes the thermal properties of the grind (Rentsen 2010). In wheat DDGS, Opoku et al. (2009) reported that steam addition has a significant effect on the bulk density and durability of the DDGS pellets. Pelleting the DDGS with a die size of 6.35 mm with steam addition produced the highest pellet bulk density (528.9 kg/m3) and durability (92.7%) as compared to a test using a 7.9 mm die size with or without steam addition. Steam conditioned DDGS, when pelleted, produced the highest bulk density using both die sizes in comparison to the pellets produced without steam addition. There was a total increase of 45.7% in the bulk density from the initial bulk density value (Tumuluru et al. 2010).

Pellet die geometry is one of the most important processing parameters in the production of pellets. The feed is mashed and compressed by rotating rollers into the die openings and is then compacted to form pellets. The die geometry is defined as the die length to diameter ratio (l/d) for each opening. Die geometry is important because as the feed passes through the die, friction produces heat, which leads to improve particle binding during compaction (Rosentrater 2007). In wheat DDGS, Opoku et al. (2009) observed that l/d ratios of 7.31 and 4.1 produced pellets with mean durabilities of 92.6 and 87.2%, respectively. In corn DDGS, l/d ratios of 9.35 and 9.0 produced pellets with durabilities of 88.34% and 21.04%, respectively (Rosentrater 2007). In another study by Rosentrater (2007), a commercial pellet mill was used to produce pellets in which l/d ratios of 10.2 and 15.3 resulted in pellets with mean durabilities of 93.93% and 88.87%, respectively.

DDGS pellets were produced at a die speed of 316 rpm (Tumuluru et al. 2010). The maximum and minimum bulk density of 0.50 g/cm^3 and 0.33 g/cm^3 were observed at 25% moisture content and 130 rpm; and 25% moisture content and 160 rpm, respectively (Chevanan et al. 2008).

2.9 Quality properties of DDGS pellets

This section of the chapter discusses the physical properties of the densified products (pellets), including density, hardness and durability.

2.9.1 Density

In wheat DDGS, Opoku et al. (2009) reported that increasing the moisture content of DDGS meal decreased the pellets bulk density. A moisture content of 11.83% (w.b.) produced the highest pellet bulk density of 514.2 kg/m^3 in comparison to a density of 436.8 kg/m^3 from a moisture content of 19.74% (w.b.). In contrast, Rosentrater (2007) found that for corn DDGS, an increased moisture content increased the pellet bulk density. Moisture contents of 5.30% and 7.65% (w.b.) produced bulk densities of 569.61 and 610.55 kg/m^3, respectively. However, the unit density decreased with an increase in moisture content. The same moisture contents produced pellets with unit densities of 1017.82 kg/m^3 994.76 kg/m^3, respectively. Xu et al. (2008) found that the unit density increased when the temperature increased from 100 to 110°C under constant pressure and then decreased by increasing the temperature further to 120°C.

2.9.2 Durability

The durability of pellets is defined by the pellet durability index (PDI). It is a measure of how resistant pellets are to abrasion and breakage (Rosentrater 2007) and will determine how resistant the material is to impact forces and vibrations during transportation and handling. Durability of

the densified product depends on various factors including moisture content, heating temperature and pressure.

In corn-based DDGS, studies have shown that increasing the moisture content from 15 to 25 % (w.b.) resulted in a 28.2% increase in durability (Chevanan et al. 2008). In a test conducted by Rosentrater (2007), the durability increased with a decrease in moisture content. In wheat DDGS pellets, a range of moisture content from 11 to 16% (w.b.) was used and the durability ranged from 60% to 93% (Opoku et al. 2009). Another test on wheat-based DDGS used moisture contents ranging from 5.10 to 11.80% (w.b) and resulted in the durability index ranging from 91.4 to 99.9 % (Tumuluru et al. 2010).

2.10 Emissions of various biomass pellets

There is an increasing interest worldwide for sustainable energy production, and bio-fuels as a renewable energy source and carbon dioxide as a neutral energy source. Biomass fuels (e.g. pellets, briquettes and powder) have become more common and fuel pellets are especially well-suited for the residential biomass market by providing possibilities for more automated and optimized systems with higher combustion efficiency and fewer products of incomplete combustion (Boman et al. 2004). In the United States, the potential benefits and challenges of biofuels are improved energy security (i.e., domestic supply, distributed resources, petroleum reduction, etc.), economic productivity (i.e., price stability, increased rural development, etc.) and environmental impacts (i.e., land and water use, greenhouse gases, carbon sequestration, etc.) (Hoekman 2009). Currently, biofuels are a small burden for water and land usage, however if bio-fuel production would significantly increase, there would be a greater demand for water

and land depending on where the crops are grown. In a study comparing the burning of fossil fuels to bio-fuels, biofuels were shown to be more environment-friendly (Demirbas 2004).

When a fuel is burned, carbon, hydrogen, nitrogen, sulphur, oxygen, ash content, volatile matter and fixed carbon are released. When compared to fossil fuels (i.e., coal), biomass fuels have lower levels of carbon and mineral ash, and higher levels of alkali and volatile products (Demirbas 2004). It has been reported that softwood pellets are an environmentally friendly fuel with low emissions (i.e., no serious effect on the environment) (Boman et al. 2003; Olsson et al. 2003; Olsson and Kjällstrand 2004; Johansson et al. 2003). Olsson (2006) found that wheat straw and peat/wood pellets have relatively low emissions during combustion. However, wood pellets burned more efficiently and with even lower emissions than straw and peat/wood pellets during flaming burning. Emissions of the polycyclic aromatic hydrocarbons naphthalene and phenanthrene were higher from straw and peat/wood pellets (Olsson 2006).

An emission study on flax shives and wood pellets showed that there was a significant difference between flax shive pellets and commercial wood pellets on emission measurements. Low emissions of methane (1.29 ppm) and oxygen (164.3 ppt) were found from the combustion of flax shive pellets. These emission values were relatively lower than the methane (1.63 ppm) and oxygen (176.6 ppt) levels measured from the combustion of commercial wood pellets. The other emissions including nitrogen, nitrogen oxide, and carbon dioxide were higher for shive pellet combustion than those for wood pellets (Rentsen 2010). Ag Fuel & Feed, a company in Indiana, has developed 100% DDGS pellets (i.e., without any addition of binders) and have used them to test burn by mixing 10% DDGS pellets with 90% coal. The DDGS pellets contained an energy level of 8,400 BTU, similar to sub-bituminous coal.

The power plant burned bituminous coal at a higher BTU level. The fuel blend diluted the power output however; the test appeared to reduce emissions. With the fuel mixture, there was a reduction in the opacity percentage, which is a measurement of visual emissions from the plant. It appears that 100% DDGS pellet caused the precipitator to perform better (Ag Fuel & Feed 2010)

2.11 Economics of pelleting

Canada produces 22 to 24 million tonnes of wheat per year with the majority coming from Saskatchewan. Fourteen to 17 million tonnes of wheat crops are exported, 2.6 to 2.9 million tonnes are used for food domestically, and 3.6 million tonnes are used for feed and dockage with carry over of 4 to 5 million tonnes. Canadian wheat-based ethanol production is based in Western Canada. The dry-milling process of ethanol production from wheat produces nearly 365 litres of ethanol, 290 kg of DDGS and 290 kg of carbon dioxide from each tonne of grain. The price of DDGS is 150-200 USD/tonne. Western Canada has seven fuel ethanol processing plants with an annual fuel ethanol production of 502 million litres (Canadian Renewable Fuels Association 2010).

In the United States, corn is used to produce ethanol. The number of corn ethanol plants has increased immensely in recent years due to the U.S renewable fuel standard. At the beginning of 2009, 193 ethanol manufacturing plants had a production capacity of nearly 46.86 billion litres per year (Rosentrater and Kongar 2009).

Distillers' dried grains with solubles are readily available all year with some dry-mill distillers processing 50-250 thousand bushels of grain per day. For each 1,000 bushels of grain, 8-9 tonnes of distillers dried grains with solubles (DDGS) are produced (Distillers Grain Technology

Council 2010). Over the years, one of the most persistent problems for effective utilization of DDGS has been its storability and flowability because these directly impact the ability to ship DDGS. DDGS is typically shipped in trains and trucks throughout North America but it is often difficult to unload once the vessel reaches its destination because the particles lock together. This results in manual unloading, which is a financial burden for the manufacturing plants, as they have to bear the labor expenses and cost of rail car repairs. Another factor is the increasing cost of conventional fuels. The cost of transporting DDGS in granular form is increasing. Rail cars or trucks containing DDGS are filled to volumetric capacity for shipping, and are often not at maximum allowable weight due to the low bulk density of the granular material itself. This wasted capacity is an additional economic loss. To alleviate DDGS flowability problems, increase the capacity of the vessels and reduce the cost of transportation, the bulk density of DDGS should be increased. Pelleting of material is one method increase the bulk density. It is a manufacturing process that is commonly used to densify granular materials, alter particle size and shape, and ultimately, product flowability and storage characteristics (Rosentrater and Kongar 2009; Opoku et al. 2009).

A biomass pellet testing performed by Mani et al. (2006c) consisted of the following processes: drying, size reduction, densifying, cooling, screening, and warehousing. Capital and operating cost of the pelleting plant was estimated. Pellet production cost for a base plant capacity of 6 tonne/h was about $51/tonne of pellets. The raw material cost was the largest element of the total pellet production cost followed by personnel cost, drying cost, and pelleting mill cost. An increase in raw material cost substantially increased the pellet production cost. Pellet plants with a capacity of more than 10 tonne/h decreased the costs to roughly $40/tonne of pellets (Mani et

al. 2006c). The cost of a pellet mill for domestic purposes is between 1000 and 4000 USD with a capacity of 150 to 200kg/h.

Pelleting reduces the cost of transportation and handling by eliminating caking of granular DDGS, which limits the flowability in rail cars and trucks. Caking of granular DDGS posed a challenge during unloading of the product, requiring extra machinery and tools (i.e., sledge hammers, shovels, pick axes, etc.), man-hours, expenses to unload, and railcar downtime. On a yearly basis, the overall cost to pelletize corn DDGS was reduced by 72% at a scale of 1000 tonnes/d compared to 100 tonnes/d (Rosentrater and Kongar 2009).

2.12 Summary

DDGS and many other biomasses have been studied extensively as a source of biofuels. To reduce the cost of handling, transportation and storage these biomass grinds are compacted or densified. Densification of various types of biomass is affected by both raw material properties, as well as, process variables. The raw material properties such as particle size, particle size distribution, bulk and particle densities, moisture content, chemical composition (proximate analysis) and angle of internal friction have been studied in various biomass materials.

The quality of densified products made from a variety of biomass feedstocks has also been studied extensively, examining parameters such as bulk and particle density, hardness, durability, and moisture absorption. Particle size reduction of biomass materials is needed before processing them into pellets. Particle size and particle size distribution affect the material properties and binding characteristics of particles during compaction. Decreasing the particle size and moisture content results in higher bulk and particle density and more particle surface area availabe for binding particles.

A wide particle size distribution is more suitable for the compaction process. At high temperatures, natural binders, including proteins and lignins, become soft and help to bind the particles together. Process variables including pressure (load), preheating temperature, steam conditioning, die geometry, and die speed affect biomass densification.

At high pressure, the biomass components change their properties and act as a binding agent for particles. Steam conditioning contributes to the activation of natural and artificial binders by causing gelatinization at an increased conditioning temperature. Reduction of particle size of the biomass increased the specific energy required for grinding. However, increasing the preheating temperature and moisture content of the feedstocks to form pellets resulted in a decrease in the specific energy required for compression. Biomass fuels have lower levels of mineral ash, higher levels of alkali and volatile products when compared to coal. Softwood, wheat straw, DDGS and peat pellets are more environmentally-friendly fuels due to their low emissions during combustion. Developing low emission pellet combustors would have a substantial positive influence on air quality.

3. MATERIALS AND METHODS

The coarse and ground distillers' dried grains with solubles (DDGS) used in this research were obtained from two ethanol plants in Saskatchewan. This chapter outlines the methodology and equipment used to determine the raw material properties, the methods followed for determination of the properties of the solid fuel pellets. The experimental designs used for characterization of the distillers' dried grains with solubles and production of the fuel pellets is also described.

3.1 Raw materials

DDGS were obtained from two ethanol-processing plants, Noramera Bioenergy Corp., Weyburn and Terra Grain Fuels, Belle Plaine, SK. The material properties and the heat of combustion of the coarse raw material and prepared ground material were determined. Then, the ground material was processed into bio-fuel pellets using the pilot-scale pellet mill. The coarse and ground DDGS both are shown in Figure 3.1.

(a) (b) (c)

Figure 3.1 Raw Materials: (a) Noramera; (b) Terra Grain; (c) Ground Terra Grain samples

3.2 Characterization of raw materials

The DDGS from Noramera Bioenergy Corp. and Terra Grain Fuels were characterized in terms of moisture content, particle size reduction, bulk and particle density, angle of internal friction and cohesion, proximate analysis and combustion energy. The supplied product was ground using a Buhler hammer mill, shown in Figure 3.2, (Buhler Manufacturing, Winnipeg and Morden, MB) with screen sizes of 3.2 mm and 4.8 mm.

Figure 3.2 Buhler hammer mill

3.2.1 Moisture content

The moisture contents of the ground DDGS from both the plants and the pellets produced from the DDGS were determined according to ASAE Standard S358.2 (ASAE 2005). In this method, 30g samples are oven-dried for 24h at 103°C and the reduction in mass is determined. A forced convective electric oven (Blue M Thermal Products Solutions, Williamsport, PA) was used and three replicates were performed for each sample. The moisture content of DDGS sample grinds from Noramera Bioenergy Corp. was adjusted to 14% and 15.5% (w.b.) by the addition of

distilled water before producing pellets. The sample grinds from Terra Grain Fuels were left at moisture contents of 11.5% and 13.09% (w.b.). The following equation was used to determine the amount of water that was required to achieve the desired moisture content of the samples,

$$m_w = m_i \frac{M_{wf} - M_{wi}}{1 - M_{wf}} \tag{3.1}$$

where m_w = mass of water added to sample (g),

m_i = initial mass of sample (g),

M_{wf} = final desired moisture content of sample (w.b.), and

M_{wi} = initial moisture content of sample (w.b.).

The grinds from Noramera Bioenergy Corp. required surface water spraying since allowing them to equilibrate for 24 hours at room temperature made the grinds too dry to form pellets. However, the grinds from Terra Grain Fuels required no manual moisture conditioning since steam conditioning was used during the pelletization process.

3.2.2 Particle size reduction

This test determines the fineness of the materials and the geometric average particle diameter of the grinds. A Ro-Tap sieve shaker (W.S. Tyler Inc., Mentor, OH) was used, which is shown in Figure 3.3. The particle size distribution of DDGS was determined according to ANSI ASAE Standard S319.4 (ASAE 2008).

Figure 3.3 Ro-Tap sieve shakers

The DDGS grinds from both plants were put into the sieves. The stack of sieves was then arranged from the largest to the smallest openings and was placed in the shaker. The sieves had Canadian series sieve numbers of 10, 12, 20, 40, 50, 60, 70, 80, 100, 140, 200 and 270 with openings of 2.00, 1.68, 1.19, 0.841, 0.420, 0.250, 0.210, 0.177, 0.149, 0.105, 0.074 and 0.053 mm, respectively. Approximately 100g of coarse material were used and the shaker was run for 10 minutes. After sieving, the particles obtained on each sieve were weighed. The geometric mean diameter (d_{gw}) and geometric standard deviation (S_{gw}) for the log-normal distribution on a weight basis were calculated as follows

$$d_{gw} = log^{-1}\left[\frac{\sum_{i=1}^{n}(m_i\ log\bar{d}_i)}{\sum_{i=1}^{n} m_i}\right] \tag{3.2}$$

$$S_{log} = \left[\frac{\sum_{i=1}^{n} m_i(\log \bar{d}_i - \log d_{gw})}{\sum_{i=1}^{n} m_i}\right]^{\frac{1}{2}} \tag{3.3}$$

$$S_{gw} \approx \frac{1}{2} d_{gw}\left[\log^{-1} S_{log} - (\log^{-1} S_{log})^{-1}\right] \tag{3.4}$$

where d_i = nominal sieve aperture size of the i^{th} sieve (mm),

d_{i+1} = nominal sieve aperture size in next larger than i^{th} sieve (mm),

d_{gw} = geometric mean diameter or median size of particles by mass (mm),

S_{log} = geometric standard deviation of log-normal distribution by mass in ten-based logarithm, dimensionless,

S_{gw} = geometric standard deviation of particle diameter by mass (mm),

m_i = mass on i^{th} sieve (g), and

n = number of sieves +1 (pan).

3.2.3 Bulk and particle density

The bulk densities of the coarse DDGS from both plants were determined using the grain bulk density apparatus. A standard 0.5L (500g) steel cup (SWA951, Superior Scale Co. Ltd., Winnipeg, MB), shown in Figure 3.4, was filled using a funnel. To maintain continuous flow, a thin steel rod was used. The cup was then gently leveled with a steel rod and weighed. The measurements were repeated three times for all samples.

The bulk density was calculated as

$$\rho_b = \frac{m}{V_c} \tag{3.5}$$

where, ρ_b = bulk density (kg/m^3),

m = mass of sample in the cylinder (kg), and

Vc = volume of cylinder (m^3).

Figure 3.4 Bulk density apparatus

To measure the particle density of the DDGS, a gas multipycnometer (Quantachrome Corp., Boynton Beach, Fl.), shown in Figure 3.5, was used. The particle densities of the coarse DDGS from both plants were determined. Prior to the particle density measurements, the multipycnometer was calibrated using a large spherical ball of known volume.

Figure 3.5 Gas multipycnometer

To determine the particle densities of the samples, first a reference volume of nitrogen gas was pressurized to about 117.2 kPa or 17 psi (P_1). The gas was then allowed to flow into the sample cell until a constant pressure, P_2, was reached. By measuring pressures P_1 and P_2, the volume of solid V_s was calculated by

$$V_s = V_{cell} - V_R\left(\frac{P_1}{P_2 - 1}\right) \tag{3.6}$$

where V_s = volume of solid (cm^3),

V_{cell} = volume of the cell (cm^3),

V_R = reference volume for the large cell (cm^3),

P_1= pressure reading after pressurizing the reference volume (kPa) and

P_2= pressure reading after including volume of the cell (kPa).

After determining the volumes for all of the samples, their masses were determined. The particle densities of the DDGS were calculated using

$$\rho_t = \frac{m}{V_s} \tag{3.7}$$

where ρ_t = particle density (kg/m^3),

m = mass of sample in the cylinder (kg), and

V_s = volume of solid (m^3).

An average particle density was obtained from three trials.

The porosity of the DDGS samples were calculated using

$$\varepsilon = \left(1 - \frac{\rho_b}{\rho_t}\right) \times 100 \tag{3.8}$$

where ε = porosity (%),

ρ_b = bulk density (kg/m^3) and

ρ_t = particle density (kg/m^3)

3.2.4 Angle of internal friction and cohesion

The frictional properties of coarse DDGS samples on a steel surface were determined in this study using the Wykeham Farrance shear box apparatus (Wykeham Farrance International Ltd., Slough, UK), shown in Figure 3.6. It was equipped with a 100 mm square shear box and motor assembly and was used to determine the angle of internal friction and cohesion of the DDGS samples. The top and bottom boxes of the apparatus were filled with DDGS and then, the bottom box was pulled horizontally at a constant speed of 0.4 mm/min.

Shear stresses using four different normal loads of 100, 200, 300, and 400 N were applied to the DDGS via a load hunger and were measured in three replicates. Calculation of the coefficient of internal friction and cohesion of the DDGS samples followed the method explained by Tabil and Sokhansanj (1997), and used

$$\tau = C_c + tan\ \varphi_i\ \sigma \tag{3.9}$$

where τ = shear stress (kPa),

C_c = cohesion (kPa),

φ_i = angle of internal friction; and

σ = normal stress (kPa).

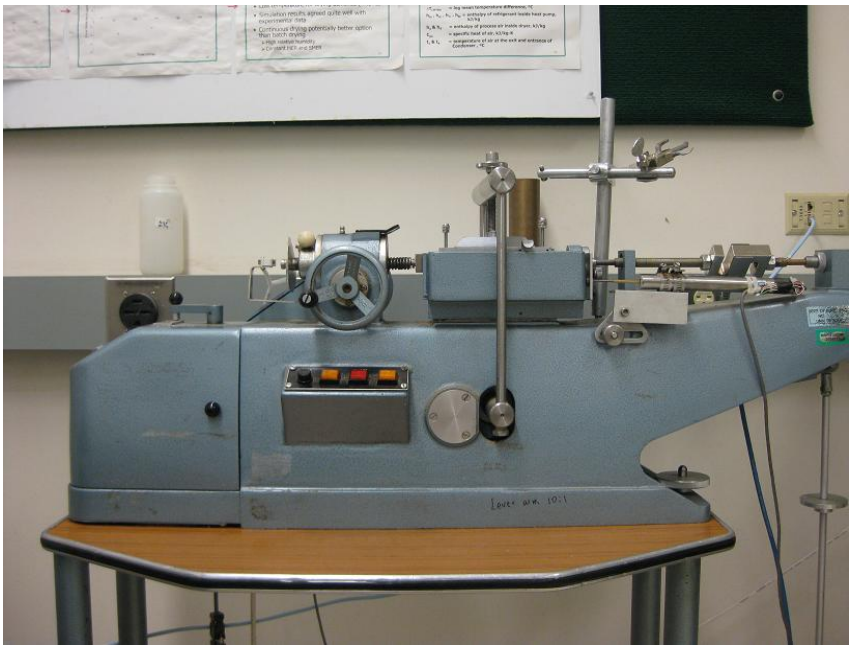

Figure 3.6 Wykeham Farrance shear box apparatus.

3.2.5 Proximate analysis

The proximate analysis of the DDGS from Noramera Bioenergy Corp. and Terra Grain Fuels was performed at the Feed Innovation Institute of the College of Agriculture and Bioresources, University of Saskatchewan to determine the amounts of dry matter, crude protein, crude fibre, cellulose, hemicellulose, lignin, starch, fat and ash contents of the samples. The methods followed were the AOAC official method 984.13 (Copper Catalyst Kjeldahl method), α amylase/amyloglucosidase method with megazyme total starch assay kit, the AOAC official method 923.03 and the AOAC official method 920.39 for determining the crude protein, starch, ash and fat (ether) contents, respectively. The amounts of acid detergent fibre (ADF), neutral detergent fibre (NDF) and acid detergent lignin (ADL) were determined using the ANKOM 200/220 Fibre Analyzer (ANKOM Technology, Macedon, NY). Cellulose and hemicellulose contents of the samples were calculated by

$$Cellulose = ADF\text{-}ADL \tag{3.10}$$

$$Hemicellulose = NDF\text{-}ADF \tag{3.11}$$

The values for the proximate analysis of the DDGS samples are expressed by weight in percentage dry basis (d.b.).

3.2.6 Combustion energy

The energy content of the DDGS was evaluated according to ASTM Standard E711-87 (ASTM 2004), the accepted test method for the combustion energy of coal and coke. The experiment was conducted at the Feed Innovation Institute of the College of Agriculture and Bioresources, University of Saskatchewan. The energy content for the samples was calculated using

$$H = \frac{(t\,W - e)}{m_a} \tag{3.12}$$

$$H_g = \frac{(t\,W - e)}{m} \tag{3.13}$$

where W = energy equivalent of calorimeter (J/°C),

H_g = energy content of standard benzoic acid (J/kg),

m_a = mass of benzoic acid (kg),

t = corrected temperature rise (°C),

e = correction for heat of firing fuse wire (J),

m = mass of sample in combustion cap (kg) and

H = energy content of sample (J/kg).

3.3 Pellet producing apparatus and method

After the DDGS samples were tested for raw material properties, the pelleting process was conducted with and without steam conditioning using the pilot-scale mill, California Pellet Mill (CPM-Laboratory Model CL-5, California Pellet Mill Co., Crawfordsville, IN), shown in Figure 3.7. The CPM has a motor power 1.5 kW (2.0-hp) and a die and roller assembly consisting of two main parts: a die, with 6.4 mm holes and a length of 46.8 mm; and a roller. The moisture content was determined prior to pelleting and moisture conditioning was done, as needed, using distilled water. The surface water was mixed into the samples using a cement mixer.

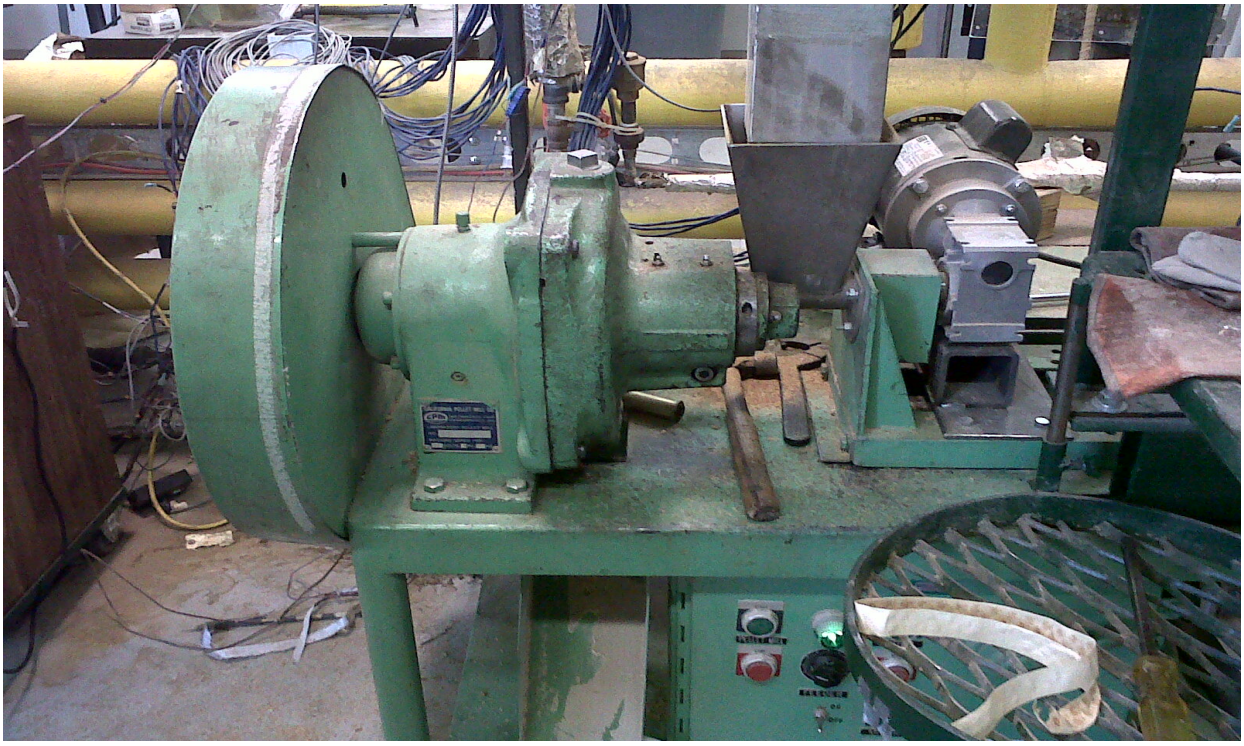

Figure 3.7 California pellet mill.

To produce pellets from Noramera Bioenergy Corp., 1.9 kg of ground DDGS was placed in the vibratory feeder and the feeder chute controlled the flow rate. To produce pellets from Terra Grain Fuels, approximately 2.5 kg of ground DDGS samples were steam conditioned and feed rate was controlled. For the samples from both plants, pellets were produced using heater

temperatures of 90 and 100°C. The temperature of the steam was maintained at 110°C and the pressure in the steam supply line were 144.78 kPa. To make the pellets, 1.474 kg/h of steam was required.

In the pelleter, the die and roller rotate in opposite directions, which creates frictional heating by high pressure and force. Plugging of the ring die and roller assembly resulted when there was too much material to be compressed through the die holes or the material was too wet. The ground DDGS were densified through the open-ended cylindrical die from the inside of the ring towards the outside of the ring and compacted particles were formed. These were compressed against each other. After pelletization, the pellets were cooled at room temperature. The pellets formed from both plants are shown in Figure 3.8.

(a) (b)

Figure 3.8 DDGS pellets (a) Noramera samples; (b) Terra Grain samples

Experiments were performed on the pellets to examine the effects of moisture content, particle size and pelleting temperature on the properties of the pellets. The quality of the pellets, in terms of bulk and particle densities, dimensional stability, hardness, durability, ash content and moisture absorption, were studied using a Full Factorial Design (FFD).

Full factorial designs measure response variables using every treatment (combination of the factor levels). A full factorial design for n factors with $N_1 ... N_n$ levels require $N_1 \times ... \times N_n$ experimental runs, i.e., one for each treatment. The design is advantageous for separating individual effects. However, full factorial designs can make large demands on data collection (Mathworks 2010). The three factors that varied were screen size, moisture content and pelletization temperature from both Noramera Bioenergy Corp. and Terra Grain Fuels. To grind the DDGS, the hammer mill used screen sizes of 3.2 and 4.8 mm. The Noramera samples had moisture contents of 14 and 15.5% (w.b.) and the Terra Grain samples had 13.09 and 11.5% (w.b.). The heating temperature was 90 or 100°C. A total 16 experimental runs were specified according to the FFD. These are shown in Table 3.1.

Table 3.1 Full Factorial Design (FFD)

Noramera Bioenergy samples without steam conditioning			
Run	Screen Size (mm)	Moisture Content (% w.b.)	Temperature (°C)
1.	3.2	15.5	100
2.	3.2	14.0	100
3.	3.2	15.5	90
4.	3.2	14.0	90
5.	4.8	15.5	100
6.	4.8	14.0	100
7.	4.8	15.5	90
8.	4.8	14.0	90
Terra Grain Fuels samples with steam conditioning			
1.	3.2	13.09	100
2.	3.2	11.5	100
3.	3.2	13.09	90
4.	3.2	11.5	90
5.	4.8	13.09	100
6.	4.8	11.5	100
7.	4.8	13.09	90
8.	4.8	11.5	90

3.4 Testing of pellet properties

To study the physical and fuel properties of the pellets formed from DDGS samples, many experimental tests were performed including tests for bulk and particle density, dimensional stability, durability, hardness, ash content, water absorption and combustion energy. To compare the emission values of the DDGS pellets, commercial wood pellets were also considered.

3.4.1 Densities of pellets

The bulk density of the pellets was determined using the grain bulk density apparatus and particle density and porosity were determined using the gas multipycnometer. The same methods were used as described earlier in the chapter for the raw coarse material. For each test, three replicates were done. Approximately 85 grams of pellets were used to determine the particle density of pellets and three replicates were performed. The porosity of the pellets was also calculated.

3.4.2 Dimensional stability

To test the dimensional stability, 10 randomly selected pellets from each experimental run of Noramera Bioenergy Corp. and Terra Grain Fuels DDGS were used. The length and diameter of the individual pellets were measured after cooling and the length/diameter ratio was calculated. Pellets from all experimental runs were sealed in bags and stored at room temperature for 21 days. The length and diameter measurements were taken again and changes in the length, diameter and length/diameter ratio of individual pellets were calculated in terms of percentage.

3.4.3 Durability

Durability of pellets was determined by a durability tester for pellets and crumbles (ASAE S269.4 DEC1991), which is shown in Figure 3.9. The durability of pellets was determined by placing a 500 g sample of pellets in the tumbling device, with a dust tight enclosure, for 10 min at 50 rpm.

Figure 3.9 Durability tester for pellets and crumbles

After tumbling, the sample was removed; sieved using Sieve size no. 4 (i.e., 4.8 mm openings) and the percentage of whole pellets was calculated. The experiment was performed three times. The pellet durability index (PDI), or durability, was calculated using

$$Durability\ (\%) = \frac{Mass\ of\ pellets\ after\ tumbling}{Mass\ of\ pellets\ before\ tumbling}\ X\ 100 \tag{3.14}$$

3.4.4 Hardness

The hardness of the DDGS pellets was determined by a compression test (ASAE standard S368.4) using the Instron universal testing machine (Model 1011, Instron Corp., Canton, MA). The hardness test assembly is shown in Figure 3.10. A horizontal flat plate, with a diameter of 57.2 mm, applied a force with a crosshead speed of 10 mm/min. This is identical to the method

used by Tabil (1996). The measurements were performed on ten randomly selected pellets from each of the 16 runs. The maximum force in Newtons (N) required to break the pellets was determined and used as a measure of the hardness.

Figure 3.10 (a) Hardness Test Assembly

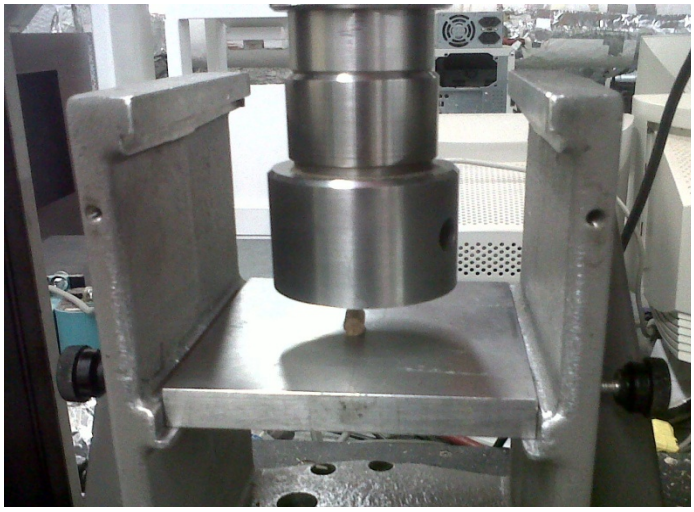
Figure 3.10 (b) Hardness test assembly (closer view)

3.4.5 Moisture absorption

Fifty gram pellet samples from each experimental run were subjected to a moisture absorption test using the temperature and humidity conditioner oven (Model AH-213, BRYANT Manufacturing Associates, Ayer, MA) shown in Figure 3.11. The pellets were placed in the oven

for three days at a temperature of 25°C and humidity of 90%. The mass of the samples was determined before placing them in the humidity conditioner oven. Each sample was measured every 24h for three days to determine the percentage change in moisture content of the samples. This experiment was replicated three times.

Figure 3.11 Temperature and humidity conditioner oven.

3.4.6 Ash content

The ash content of the DDGS pellets was determined using the standard test method for ash in wood (ASTM D1102-84 2007). The ash percentage was determined using the muffle furnace (Lab heat Muffle Furnace, Blue M Electric Company) shown in Figure 3.12. Porcelain crucibles with covers and two gram test specimens were used. First, the crucible was weighed alone and then the crucible with the sample was weighed and placed in a drying oven at 103°C. After two days, the samples were weighed again and the crucibles with covers removed were placed in the muffle furnace at 600°C. The contents were ignited until all the carbon was eliminated. The crucibles were then moved to a desiccator. The covers were replaced loosely and the crucibles were cooled and weighed. This experiment was replicated three times for each of the DDGS samples.

The percentage of ash was calculated as

$$Ash, \% = (W_1/W_2) \times 100 \quad (3.15)$$

where; W_1 = mass of the ash, and

W_2 = mass of oven-dry sample.

Figure 3.12 Muffle Furnace

3.4.7 Combustion energy

The combustion energy of individual biomass pellets produced by the California pellet mill was measured using the bomb calorimeter as discussed previously. The experiment was conducted at the Feed Innovation Institute in the College of Agriculture and Bioresources, University of Saskatchewan. The experiment was performed three times on DDGS sample pellets from each experimental run.

3.4.8 Emission values

Emission tests of the fuel pellets were performed using the Prairie Fire Stove (Model # PFG 060, Prairie Fire Grain Energy Inc., Bruno, SK) shown in Figure 3.13. Approximately 2 kg samples of pellets produced using DDGS from both plants Noramera Bioenergy Corp. and Terra Grain Fuels were used for the emission measurements. For comparison purposes, commercial wood pellets were also tested. A small quantity of pellets was placed in the firebox (Figure 3.14a) to start the fire.

For burning of the fuel pellets, by setting the fuel control knob at the middle level, the feed rate was controlled and the temperature control knob was set at a position between the low and high levels. Until the temperature inside the stove rose, it was necessary to press the prime button continually for a few minutes to start the feeding auger. After 25-30 minutes of burning, the combustion process was assumed to be in a steady state and the first gas sample was taken using a needle and syringe at the outlet shown in Figure 3.14(b).

Seven samples were taken at the interval of two minutes to evaluate the emissions. This evaluation was conducted using a gas chromatography (GC) analysis in the Department of Soil Science at the University of Saskatchewan.

Figure 3.13 Grain burning stove used to measure emissions.

(a) (b)

Figure 3.14 Components of the grain burning stove (a) Firebox, (b) Outlet where gas samples were collected

Nitrogen (N), oxygen (O_2), methane (CH_4), nitrous oxide (N_2O), and carbon dioxide (CO_2) were measured and analyzed. The detector used for N_2O and CH_4 was the Varian CP-3800 gas chromatograph shown in Figure 3.15. Nitrous oxide is detected with one of two electron capture detectors (ECD). The columns used were Poraplot Q and coated plot fused silica. They were 32.80 m in length and 0.32 mm in diameter with a film thickness of 0.32µm.

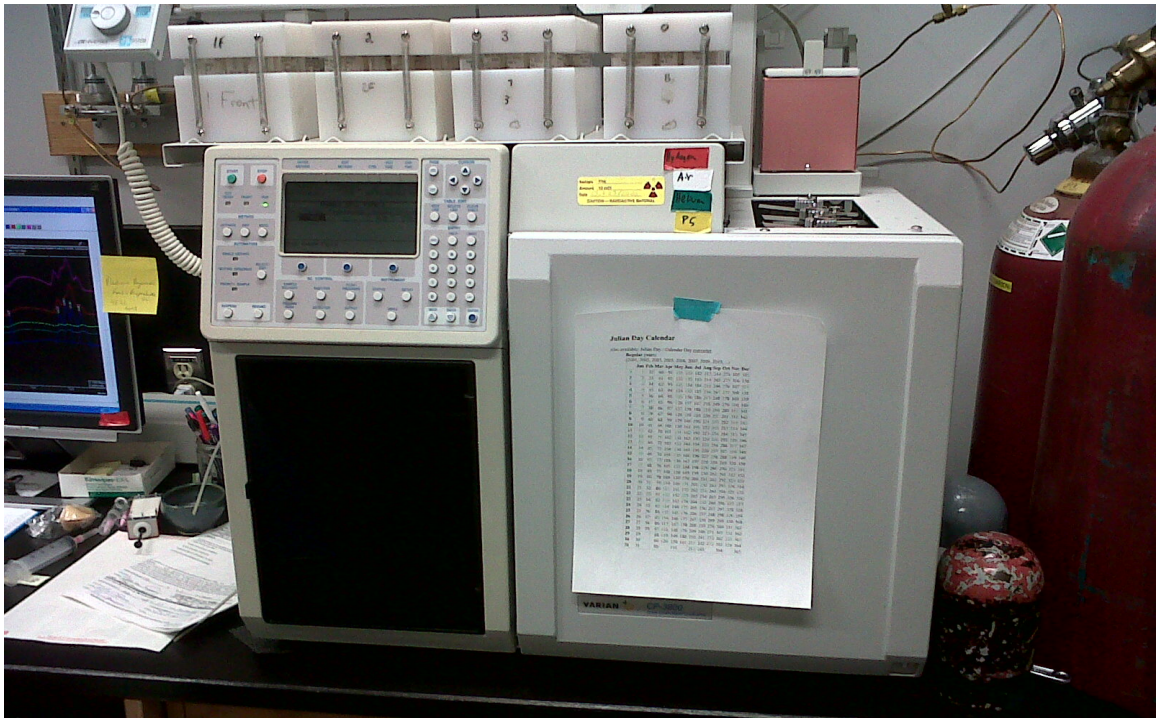

Figure 3.15 Varian CP-3800 Gas chromatograph

The detector used for methane is a flame ionization detector. The column used is a Porapak Q8, which is 3.65 m in length, 3.175 mm in diameter with 2 mm film thickness. The detection limit for methane is 360 ppb. The carrier gas for all detectors is helium and the make-up for the ECD is argon with 5% methane, also called P5. The detector used for CO_2 analysis is a Varian Micro-GC CP-2003 shown in Figure 3.16.

The CO_2 is identified using a TCD (thermal conductivity detector). The column used is a Poraplot U, which is 10 meters in length and 0.32 mm inner diameter. The detection limit for CO_2 is 80 ppm. Nitrogen (N) and oxygen (O_2) were identified using a TCD and a molecular sieve column 10 meters in length.

Figure 3.16 Varian Micro-GC CP-2003

3.5 Statistical Analysis

SPSS 14.0 statistical software for Windows (SPSS Inc., Chicago, IL) was used for the analysis of the data collected. The DDGS pellets produced with material from both plants were analyzed using the SPSS General Linear Model (GLM) univariate function for determination of analysis of variance (ANOVA) on the basis of the three variables; moisture content, screen size (particle size of DDGS) and temperature of production; and their interactions with dependent variables like stability, density, hardness, etc. The GLM includes estimation procedures for parameters in

models with a wide range of error distributions. Additional investigations missed with ANOVA were examined using linear regression and the enter method. For the normal distribution of errors, ANOVA was used which is based on mean square estimation procedures. The standard errors and standard deviations were calculated using Windows Microsoft Excel 2007.

4. RESULTS AND DISCUSSION

This chapter includes the results of the experiments described in the previous chapter. The material properties of the DDGS (distillers' dried grains with solubles), their chemical composition, and combustion energy are presented first. Following the discussion of the raw materials, results regarding pellet properties, such as dimensional stability, density, durability, hardness, ash content and moisture absorption, are presented. Lastly, the combustion energy and emissions of DDGS pellets are compared with commercial wood pellets.

4.1 Material properties of DDGS

DDGS were obtained from two plants in Saskatchewan, Noramera Bioenergy Corp. and Terra Grain Fuels Ltd. The physical properties measured were moisture content, particle size, bulk and particle density, and angle of friction were measured. Some of the properties were determined for both the ground and unground samples.

4.1.1 Moisture content

The initial moisture content of the coarse/unground DDGS samples from Noramera and Terra Grain were 12.5% and 13.75% (w.b.), respectively. The mean moisture content of DDGS after two grinding treatments is given in Table 4.1.

Table 4.1 Moisture contents of DDGS used for pelletization

Screen size (mm)	Moisture content (% w.b.)*	
	Noramera	Terra Grain
Unground	12.50 (0.05)	13.75 (0.07)
4.8 mm	12.03 (0.04)	13.09 (0.07)
3.2 mm	11.60 (0.07)	11.50 (0.06)

*Numbers in parenthesis are standard deviations (n=3)

The results show that, after grinding with a hammer mill, the moisture content of the samples decreases. This indicates that more heat is generated during grinding using a smaller screen size resulting in those samples losing more moisture.

4.1.2 Particle size

Particle size is a very important material property as it affects the grinding process and production of the pellets. Therefore, particles sizes of the DDGS were determined in this study. The geometric mean diameter (d_{gw}) of particles for unground and ground DDGS (hammer mill screen size 3.2 & 4.8 mm) from both sources, along with the geometric standard deviation (S_{gw}), are given in Table 4.2.

Table 4.2 Geometric mean diameter (d_{gw}) and standard deviation (S_{gw}) of DDGS

Screen Size	Noramera		Terra Grain	
(mm)	*d_{gw} (mm)	**S_{gw} (mm)	*d_{gw} (mm)	**S_{gw} (mm)
Unground	2.49	1.57	1.46	1.36
4.8 mm	0.78	0.65	0.52	0.41
3.2 mm	0.73	0.62	0.49	0.39

*d_{gw} = geometric mean length/diameter or median size of particles by mass, mm
**S_{gw} = geometric standard deviation of particle length/diameter by mass, mm

For the coarse/unground DDGS from Noramera, the geometric mean diameter was 2.49 mm whereas, the DDGS from Terra Grain had a geometric mean diameter of 1.46 mm. The geometric standard deviations of the unground DDGS were 1.57 and 1.36 mm for Noramera and Terra Grain, respectively. Grinds from the screen size of 3.2 mm had lower standard deviations of 0.62 and 0.39 mm, respectively. For the Noramera samples, the use of hammer mill screens of 4.8 and 3.2 mm resulted in geometric mean diameters with values of 0.783 and 0.726 mm, respectively and, for Terra Grain samples, the values were 0.52 and 0.49 mm, respectively.

Figure 4.1 compares the particle size distribution after grinding of the DDGS from Noramera Bioenergy Corp. The largest amount of material was retained on the sieve with an opening of 1.19 mm for both the screen sizes. Overall, the grinds from 2.00 to 0.42 mm screens had a large size distribution. The amount of material retained in the sieves from 0.21 to 0.04 mm is very low because the more of the samples remained on the bigger screen sizes.

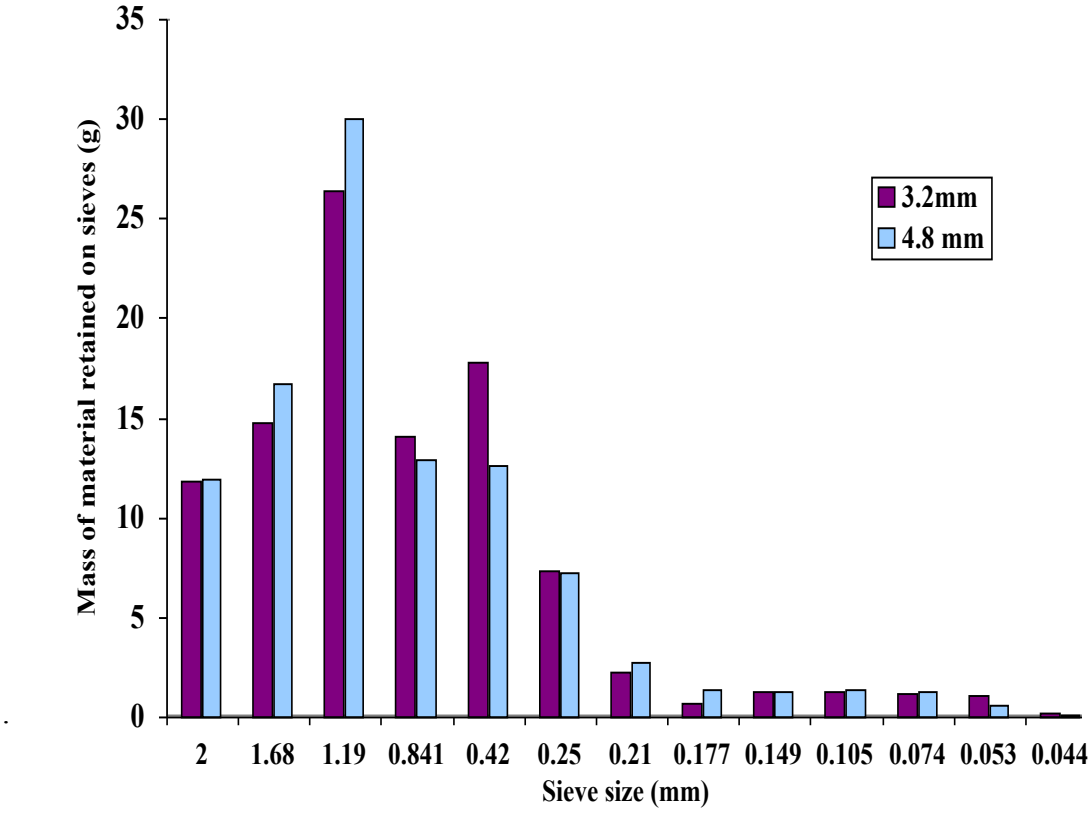

Figure 4.1 Particle size distribution of DDGS from Noramera Bioenergy Corp. at two different screen sizes.

Figure 4.2 compares the particle size distribution from Terra Grain Fuels samples after grinding. The largest amount of material was retained on the sieve with openings of 0.42 mm. A large size distribution is seen on the sieves with openings from 1.19 to 0.25 mm. Less amount of material is retained on the smaller sieves with opening sizes from 0.18 to 0.04 mm. Mani et al. (2004a)

used sieve sizes with openings ranging from 0.09 to 2.00 mm to determine the particle size distribution of various biomass grinds at various screen sizes. They found that the grinds from screen size 3.2 mm had the largest size distribution.

Therefore, it can be concluded that the sieve sizes where the largest amount of material was retained shows that the material is getting more uniform in size. In Noramera samples, the 4.8 mm screen size retained the largest amount of material whereas, in Terra grain samples, the 3.2 mm screen size retained the largest amount of material. This shows that the particles of Noramera samples were larger than the Terra grain samples.

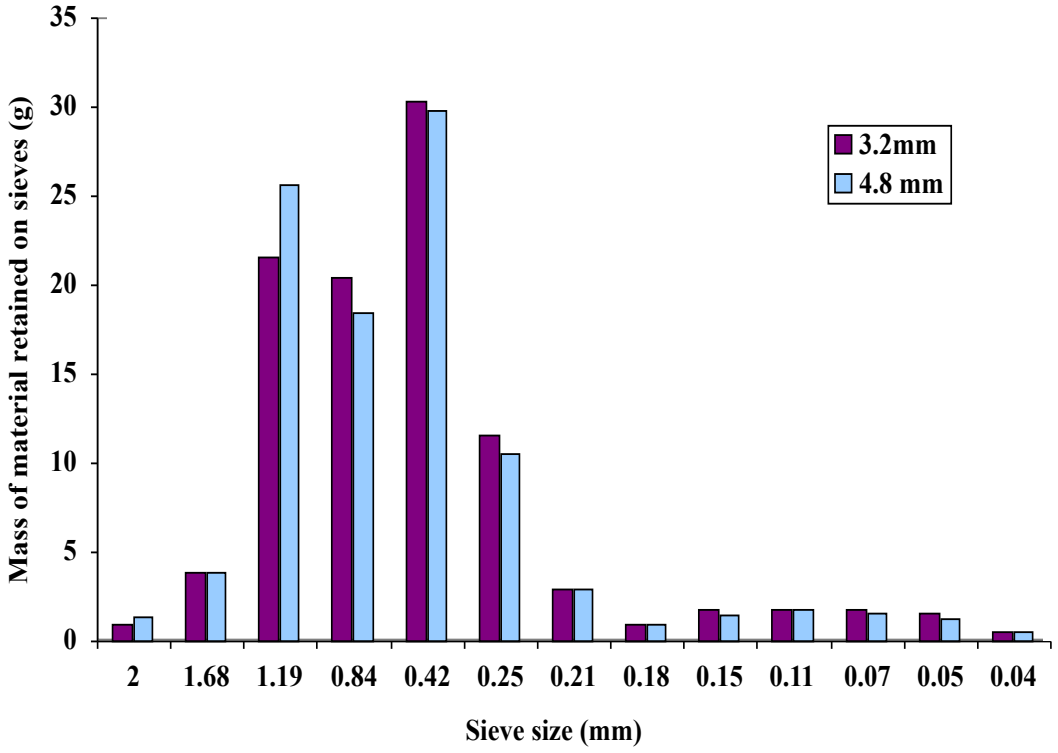

Figure 4.2 Particle size distribution of DDGS from Terra Grain Fuels Ltd. at two different screen sizes.

4.1.3 Bulk and particle density

The bulk and particle densities and porosity results from the ground Noramera DDGS are shown in Table 4.3. The initial bulk and particle densities of the coarse DDGS were determined to be, on average, 658.8 and 1135.0 kg/m^3, respectively.

Table 4.3 Means of bulk & particle density and porosity of DDGS from Noramera Bioenergy Corp.

Screen size (mm)	Bulk density (kg/m^3)*	Particle density (kg/m^3)*	Porosity (%)
4.8	687.56 (38.06)	1278 (31.43)	46.20
3.2	695.84 (4.64)	1343 (6.56)	48.19

*Numbers in parenthesis are standard deviations of densities of DDGS (n=3)

From the above results, it is shown that both the bulk and particle densities of DDGS increased as the screen size decreased. The porosity of the ground DDGS increased with a decrease in screen size. Table 4.3.1 shows the results of a statistical analysis, using ANOVA, of the affect of different screen sizes on the bulk and particle densities of DDGS from Noramera Bioenergy Corp. This analysis shows that use of a smaller screen size has a significant affect on the particle density, but not on the bulk density.

Table 4.3.1 Analysis of variance (ANOVA) of densities of ground DDGS (Noramera Bioenergy Corp.)

Source	df	Mean square	
		Bulk density	Particle density
Screen sizes	1	102.84^{ns}	6337.50*
Error	4	735.25	515.50

*$p<0.05$, ns= not significant

The initial bulk and particle densities of the coarse DDGS from Terra Grain Fuels were 451.5 and 809.4 kg/m^3, respectively. The average bulk and particle densities and porosities of the ground DDGS are shown in Table 4.4.

Table 4.4 Means of bulk and particle densities and porosity of ground DDGS from Terra Grain Fuels Ltd.

Screen size (mm)	Bulk density (kg/m^3)*	Particle density (kg/m^3)*	Porosity (%)
4.8	483.63 (5.91)	817 (7.55)	89.82
3.2	534.42 (8.21)	869 (9.85)	88.69

*Numbers in parenthesis are standard deviations of densities of DDGS (n=3)

Both, the particle and bulk densities of the ground material increased with a decrease in screen size. ANOVA Table 4.4.1 shows how the different screen sizes used for grinding DDGS from Terra Grain Fuels affected the particle and bulk densities. The analysis shows that use of a smaller screen size caused a statistically significant increase in both the bulk and particle densities of DDGS.

Table 4.4.1 Analysis of variance (ANOVA) of densities of ground DDGS (Terra Grain Fuels Ltd.)

Source	df	Mean square	
		Bulk density	Particle density
Screen sizes	1	3869.44*	4056.00*
Error	4	51.22	77.00

*$p < 0.05$

4.1.5 Angle of internal friction and cohesion

The angle of friction and cohesion results of raw DDGS samples from both the plants are given in Table 4.5. Regression equations for the angle and coefficient of internal friction and cohesion were estimated for data at four normal loads of 100, 200, 300 and 400N.

Table 4.5 Angle of internal friction and cohesion of DDGS

DDGS	μ_i	Φi (Degree)	*Cohesion C_c		
			Estimate (kPa)	R^2	SEE
Noramera	0.103	5.869	8.534	0.948	0.295
Terra Grain	0.101	5.755	7.402	0.952	0.278

*Values are averages of three replicates.

μ_i: coefficient of internal friction.

Φi: angle of internal friction.

SEE: standard error of estimate of cohesion

The cohesion value was higher in the Noramera samples (8.534 kPa) than the Terra Grain samples (7.402 kPa). This suggests that inter-molecular bonding between the particles was higher in the Noramera samples and they were more uniform. Figure B.1 and B.2 also shows that there is a linear relationship between shear stress and normal stress of the raw materials from Noramera Bioenergy Corp. and Terra Grain Fuels Ltd. There was no significant difference in the angle of internal friction in either of the samples.

The moisture content plays an important role. The moisture contents of the Noramera and Terra Grain samples were 12.5% (w.b.) and 13.75% (w.b.), respectively. A higher moisture content generally means that the coefficient of friction will be higher but, in this case, no such clear tendency was observed.

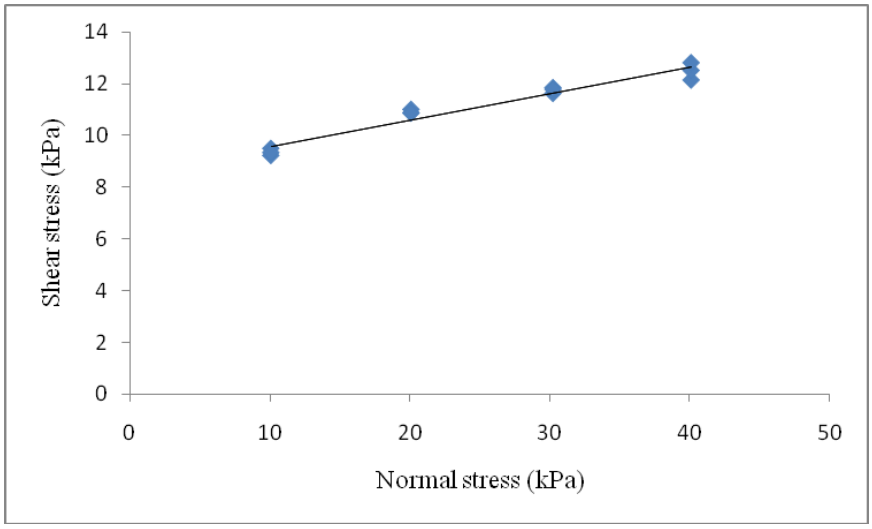

Figure B.1 Normal stress-shear plot for friction measurement of Noramera samples

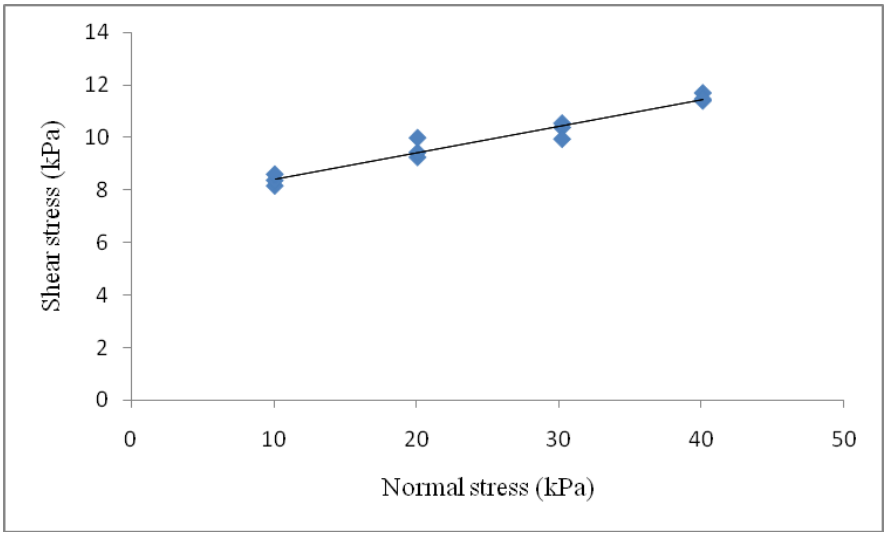

Figure B.2 Normal stress-shear plot for friction measurement of Terra Grain samples

4.2 Chemical properties

The DDGS were tested for their proximate analysis and the chemical compositions are discussed. Also, the combustion energy of the DDGS is presented.

4.2.1 Chemical composition

The chemical composition of DDGS from Noramera Bioenergy and Terra Grain Fuels are shown in Table 4.6. The moisture contents of the raw DDGS samples at the time of testing were 8.6 and 12.31% (w.b.) from Noramera and Terra Grain samples, respectively.

Table 4.6 Chemical compositions of DDGS (in percentage dry basis, % d.b.)

Raw materials	Dry matter (%)	Crude fibre (%)	Crude protein (%)	Cellulose (%)	Hemi-cellulose (%)	Lignin (%)	Starch (%)	Fat (Ether) (%)	Ash (%)
Noramera	91.40	4.98	37.41	10.75	21.04	10.50	3.84	4.52	5.16
Terra Grain	87.69	7.33	32.43	10.81	27.45	4.37	4.18	6.37	4.50

The Noramera samples had a higher percentage of lignin (10.5%) compared to the Terra Grain samples, which had (4.37%). Lignin helps in adhesion of the particles and the smaller percentage in the Terra Grain samples kept the pellets from forming without steam addition. Steam conditioning activates the starch present in the samples and helps it to gelatinize. More protein was found more in the Noramera samples, which helps to increase the durability.

4.2.2 Combustion energy

The combustion energy of the Noramera samples was 19.45 MJ/kg at a moisture content of 8.6% (w.b.), whereas the combustion energy of Terra Grain samples was 18.54 MJ/kg at 12.31% (w.b.) moisture. Earlier studies have shown that the combustion energies of poplar and wheat straw were 17.76 MJ/kg at a moisture content of 8.2% (w.b.) and 17.04 MJ/kg at a moisture content of 8.0% (w.b.), respectively (Shaw, 2008).

4.3 Effect of DDGS particle size on pellet durability

Pellets were formed from the Noramera and Terra Grain DDGS. One-way ANOVA was used to analyze the significance of screen size on pellet durability. The mean durability values of the pellets are shown in Table 4.7. The durability of the pellets increased as the screen size decreased which is presumably due to the fact that a small screen size produces more fine particles, which fill voids in the pellets, and hence makes them more durable.

Table 4.7 Durability means of pellets

Biomass Source	Screen Sizes (mm)	Durability* (%)
Noramera Bioenergy Corp.	3.2	88.74 (1.39)
	4.8	81.52 (1.16)
Terra Grain Fuels Ltd.	3.2	92.25 (1.97)
	4.8	82.69 (1.19)

* Values in parenthesis are standard error; n = 12

The results of one-way ANOVA are shown in Tables 4.7.1 and 4.7.2 for the Noramera and Terra Grain sample pellets, respectively. They show that reduction in screen size has a statistically significant affect in terms of durability of the pellets.

Table 4.7.1 Analysis of varaiance of durability of pellets produced from Noramera Bioenergy Corp.

Source of Variation	df	Mean square
Between Groups	3	52938.36*
Within Groups	92	14.93

Table 4.7.2 Analysis of variance of durability of pellets produced from Terra Grain Fuels Ltd.

Source of Variation	df	Mean square
Between Groups	3	55730.77*
Within Groups	92	20.44

* $p<0.05$

4.4 Effect of screen size, moisture content and temperature on the material properties of DDGS pellets produced from Noramera Bioenergy Corp. and Terra Grain Fuels Ltd.

ANOVA and multiple regression analysis were used to determine the effects of particle size, moisture content and temperature on the dimensional stability, bulk and particle densities, porosity, hardness, durability, and moisture absorption of the DDGS pellets produced from Noramera Bioenergy Corp. and Terra Grain Fuels materials.

4.4.1 Dimensional stability of pellets

4.4.1.1 Dimensional stability of Noramera pellets

The dimensional stability of the pellets formed was determined in terms of the change in length diameter, and ratio of length to diameter and is shown in Table 4.8.

Table 4.8 Changes in length, diameter and ratio of length/diameter of Noramera pellets

Parameters			Dimensional Stability		
Screen Size (mm)	Moisture Content (%), w.b.	Temperature (°C)	Length (%)	Diameter (%)	Length/Diameter (%)
3.2	15.5	100	0.17 (0.01)	0.05 (0.02)	0.01 (0.01)
3.2	15.5	90	0.13 (0.04)	0.04 (0.01)	0.01 (0.01)
3.2	14.0	100	0.32 (0.02)	0.06 (0.01)	0.02 (0.01)
3.2	14.0	90	0.31 (0.03)	0.05 (0.01)	0.03 (0.01)
4.8	15.5	100	0.08 (0.01)	0.02 (0.01)	0.01 (0.01)
4.8	15.5	90	0.16 (0.02)	0.04 (0.01)	0.01 (0.00)
4.8	14.0	100	0.36 (0.01)	0.05 (0.01)	0.01 (0.00)
4.8	14.0	90	0.36 (0.01)	0.02 (0.00)	0.04 (0.01)

* Values in parenthesis are standard error; n = 10

Table 4.8.1 contains the ANOVA results that show how the length, diameter, and ratio of length to diameter are affected by various factors. Experimentally, the change in length was greater for a moisture content of 14% than 15.5% (w.b.). Pellets formed from material at a higher moisture content did not absorb as much moisture, and therefore, the pellet length did not increase as much as for the lower moisture content.

Table 4.8.1 Mean square values of ANOVA of change in length, diameter and ratio of length/diameter of Noramera Bioenergy Corp. pellets.

Source	df	Length	Diameter	Length/Diameter
Screen size	1	0.000^{ns}	0.004*	0.001*
Moisture content	1	0.803*	0.000^{ns}	0.016*
Temperature	1	0.001^{ns}	0.001*	0.000*
Screen size*Moisture content	1	0.028*	0.004^{ns}	0.001*
Screen size*Temperature	1	0.019*	0.001*	0.006^{ns}
Moisture content*Temperature	1	0.003^{ns}	0.002*	0.000^{ns}
Screen size*Moisture content*Temperature	1	0.14*	0.004*	0.005^{ns}
Error	72	0.180	0.011	0.004

*p<0.05, ns=not significant.

After the factors were determined to be significant using ANOVA analysis, a multiple regression analysis was performed to illustrate the effect of the two-way interactions of moisture content and screen size on change in length (Δl) of the pellets. The regression model used which shows the effects of moisture content (X_1), screen size (X_2), and their interaction (X_1X_2) is given as

$$\Delta l\ (\%) = 0.339 - 0.008\ X_1 + 0.467\ X_2 - 0.031\ X_1X_2 \qquad (4.1)$$

Figure 4.3 shows a plot of this regression model. The graph shows that the change in pellet length was affected the most as the screen size increased and moisture content decreased.

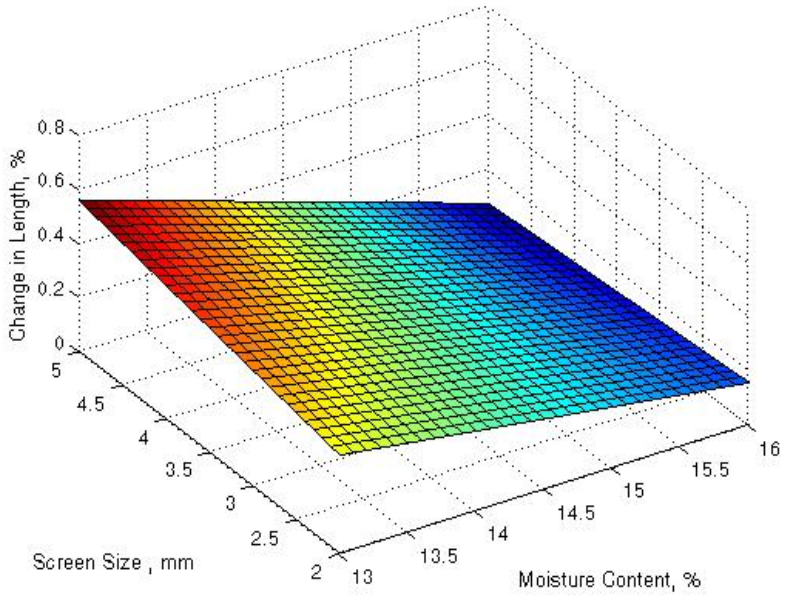

Figure 4.3 Change in pellet length showing interaction between moisture content and screen size

The results of multiple regression analysis for the interaction of screen size (X_2) and temperature (X_3) are shown in Figure 4.4 and the regression equation for the model is given as

$$\Delta l\ (\%) = -1.183+0.374\ X_2+0.015\ X_3-0.004\ X_2X_3 \qquad (4.2)$$

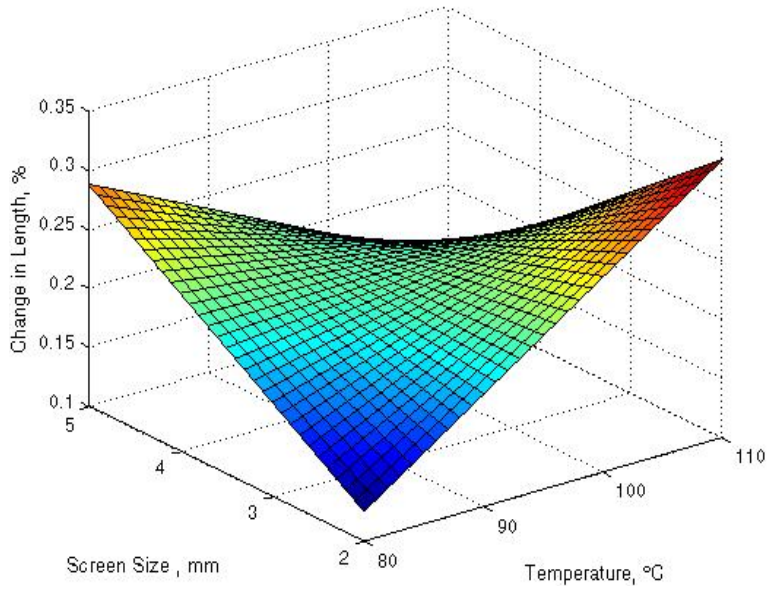

Figure 4.4 Change in pellet length showing interaction between screen size and temperature

The graph shows that essentially two maximum and two minimum length changes occurred for these parameters. The highest temperature combined with smallest screen size and lowest temperature combined with largest screen size resulted in the largest changes in pellet length. The opposite of these, i.e. lowest temperature with largest screen size and highest temperature with smallest screen size, resulted in the smallest changes in the pellet length.

The statistical analysis (ANOVA) in Table 4.8.1 showed that statistically significant change in diameter occurred due to the interactions of screen size and temperature, and moisture content and temperature. A plot of the regression model that shows the effect on the change in diameter of the interaction between screen size (X_2) and temperature (X_3) is shown in Figure 4.5 and the equation used to produce this plot is

$$\Delta d\ (\%) = -0.32861+0.076841\ X_2+0.004261\ X_3-0.0009\ X_2X_3 \qquad (4.3)$$

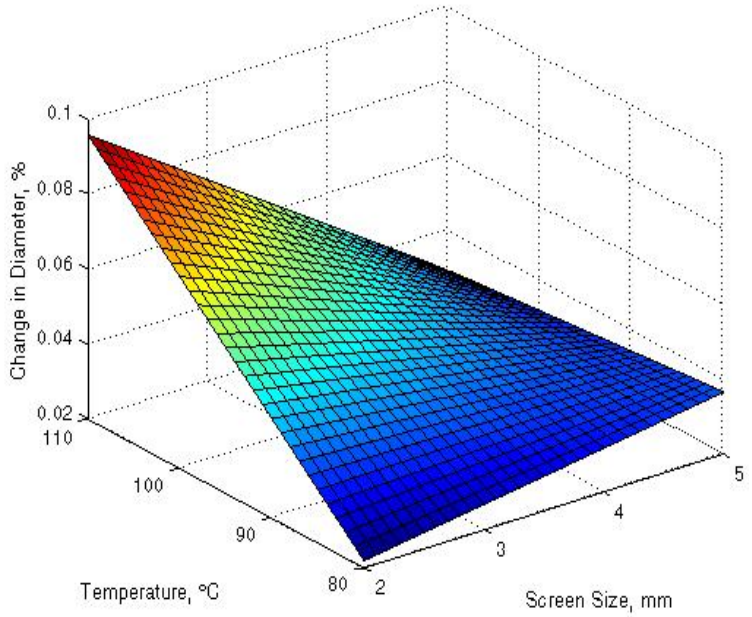

Figure 4.5 Lateral expansions of pellets showing interaction between screen size and temperature

The graph shows that the lateral expansion of pellets was greatest for the highest temperature and largest screen size, whereas the least lateral expansion occurred for the lowest temperature and smallest screen size.

A plot of the regression model showing the effect of the interaction between moisture content (X_1) and temperature (X_3) is shown in Figure 4.6 and the equation for the model is.

$$\Delta d\ (\%) = -1.77389+0.118823\ X_1+0.019543\ X_3-0.00128\ X_1X_3 \qquad (4.4)$$

The graph shows that two maximum changes in pellet diameter occurred. Lateral expansion occurred more with higher temperature and low moisture content and another change occurred with low temperature and higher moisture content. Shaw (2008) also found that pellet expansion decreased with a decrease in moisture content.

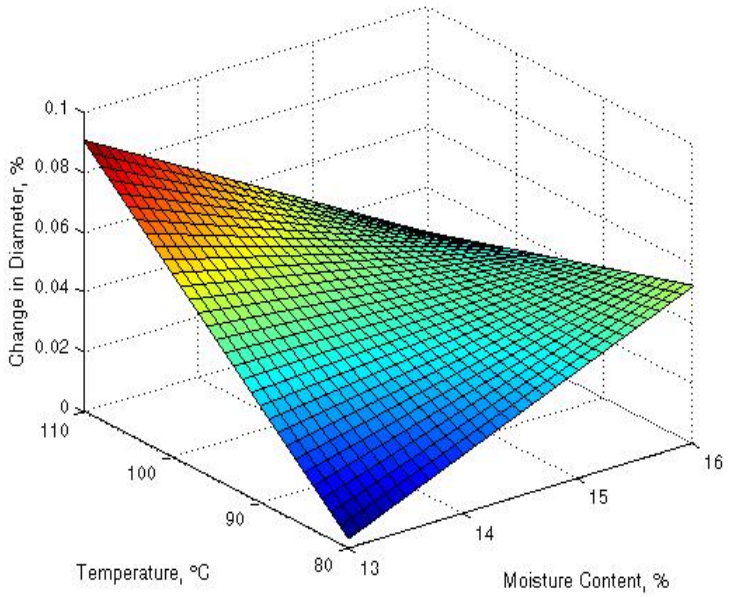

Figure 4.6 Lateral expansions of pellets showing interaction between moisture content and temperature

From the ANOVA shown in Table 4.8.1, the only significant affect on the change in length to diameter ratio was an interaction between moisture content (X_1) and screen size (X_2). A plot of

the regression model showing this interaction is given in Figure 4.7 and the regression model equation is

$$\Delta l/d\ (\%) = -0.0559795+0.00387006\ X_1+0.08714497\ X_2-0.0056151\ X_1X_2 \quad (4.5)$$

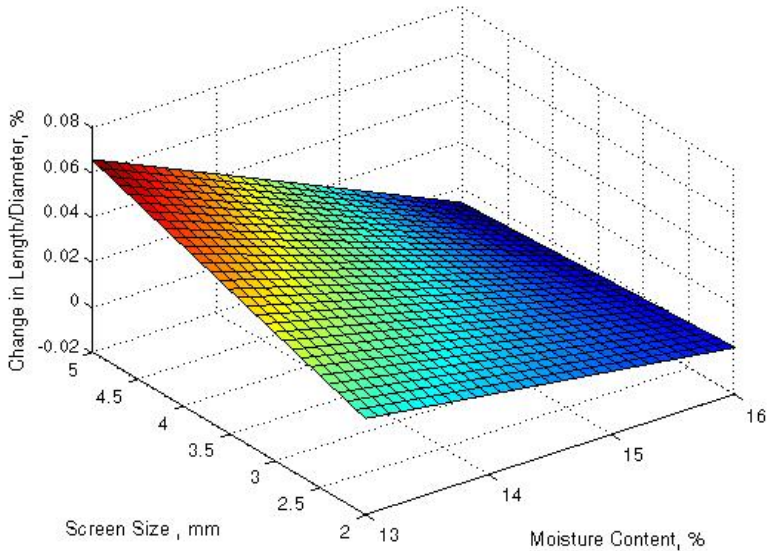

Figure 4.7 Change in length to diameter ratio showing interaction between screen size and moisture content

The trend shown in Figure 4.7 is similar to that of Figure 4.3. It clearly illustrates the effect of screen size and moisture content on the change in pellet length. This is as anticipated since the change in diameter was not significantly affected by either of these factors. Therefore, the change in the ratio is dependent only on the length change.

4.4.1.2 Dimensional stability of Terra Grain pellets

The dimensional stability of the pellets formed from the Terra Grain DDGS was determined by taking measurements regarding the changes in the length, diameter, and ratio of length to diameter. These measurements are given in Table 4.9. Table 4.9.1 contains the ANOVA results that show how the length, diameter, and ratio of length to diameter are affected by various factors.

Table 4.9 Changes in length, diameter and ratio of length/diameter of Terra Grain pellets

Parameters			Dimensional Stability		
Screen Size (mm)	Moisture Content (%), w.b.	Temperature (°C)	Length (%)	Diameter (%)	Length/Diameter (%)
3.2	13.09	100	0.12 (0.01)	0.03 (0.01)	0.01 (0.00)
3.2	13.09	90	0.19 (0.02)	0.02 (0.01)	0.01 (0.01)
3.2	11.5	100	0.45 (0.02)	0.03 (0.01)	0.04 (0.01)
3.2	11.5	90	0.35 (0.03)	0.04 (0.02)	0.04 (0.02)
4.8	13.09	100	0.08 (0.01)	0.02 (0.00)	0.01 (0.00)
4.8	13.09	90	0.07 (0.01)	0.02 (0.00)	0.01 (0.00)
4.8	11.5	100	0.34 (0.01)	0.05 (0.02)	0.03 (0.01)
4.8	11.5	90	0.40 (0.02)	0.05 (0.01)	0.03 (0.01)

* Values in parenthesis are standard error; n = 10

Table 4.9.1 Mean square values of ANOVA of change in length, diameter and ratio of length/diameter of Terra Grains Fuel Ltd. pellets.

Source	df	Length	Diameter	Length/Diameter
Screen size	1	0.009*	0.000^{ns}	0.000*
Moisture content	1	1.366*	0.007*	0.020*
Temperature	1	0.002^{ns}	0.001^{ns}	0.007^{ns}
Screen size*Moisture content	1	0.001^{ns}	0.002*	0.000*
Screen size*Temperature	1	0.003^{ns}	0.002^{ns}	0.004^{ns}
Moisture content*Temperature	1	0.000^{ns}	0.001*	0.008^{ns}
Screen size*Moisture content*Temperature	1	0.001^{ns}	0.000^{ns}	0.009^{ns}
Error	72	0.091	0.008	0.003

*$p<0.05$, ns=not significant.

After factors were determined to be statistically significant using ANOVA analysis, multiple regression analyses were done to illustrate the significant effect on the change in length (Δl) of the pellets. A plot of the regression model showing the effects of moisture content (X_1) and screen size (X_2) is given in Figure 4.8. The regression equation used is

$$\Delta l\ (\%) = 2.300961 - 0.16439\ X_1 - 0.01352\ X_2 \quad (4.6)$$

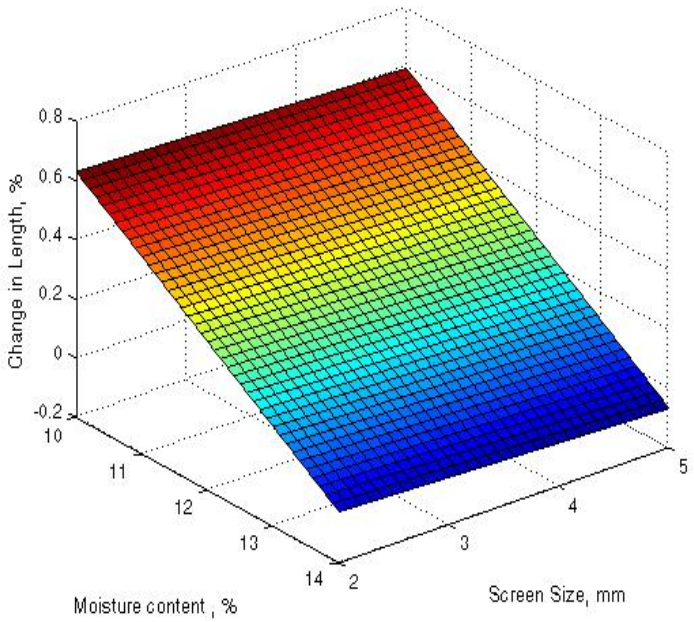

Figure 4.8 Change in pellet length at different moisture contents and screen sizes

Figure 4.8 shows that the largest change in pellet length occurred with a low moisture content and small screen size, although the affect of screen size was not significant. This agrees with the experimental results in which the largest change in pellet length occurred at a moisture content of 11.5% (w.b.) and screen size of 3.2 mm.

To study the affect on the change in pellet diameter, multiple regression analysis was performed. The regression model that shows the affects of moisture content (X_1), screen size (X_2), and their interaction (X_1X_2) is

$$\Delta d\ (\%) = -0.20237 + 0.018447\ X_1 + 0.095311\ X_2 - 0.00756\ X_1X_2 \quad (4.7)$$

Figure 4.9 is a plot of this regression equation. It shows that lateral expansion was greater with increase in screen size and moisture content.

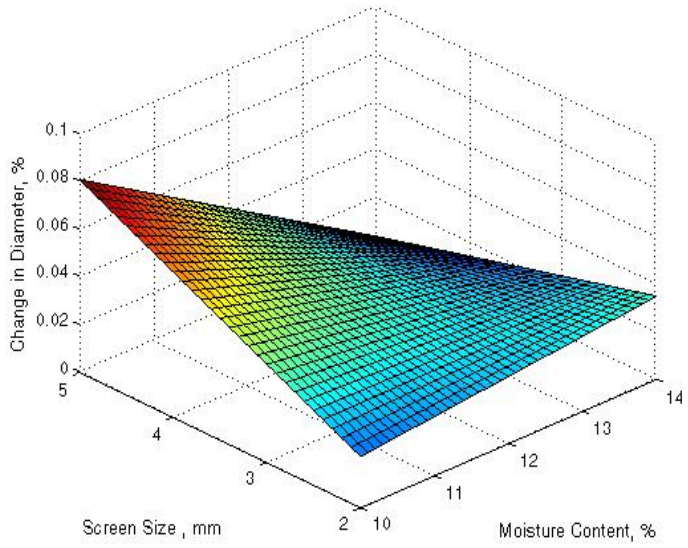

Figure 4.9 Lateral expansions of pellets showing interaction between screen size and moisture content

The regression model showing the affects of moisture content (X_1), temperature (X_3), and their interaction (X_1X_3) on change in pellet diameter is

$$\Delta d\ (\%) = 1.100519 - 0.08613\ X_1 - 0.0097\ X_3 + 0.000782\ X_1X_3 \qquad (4.8)$$

Figure 4.10 is a plot of this regression equation. It shows that lateral expansion was greater with a decrease in moisture content and an increase in temperature.

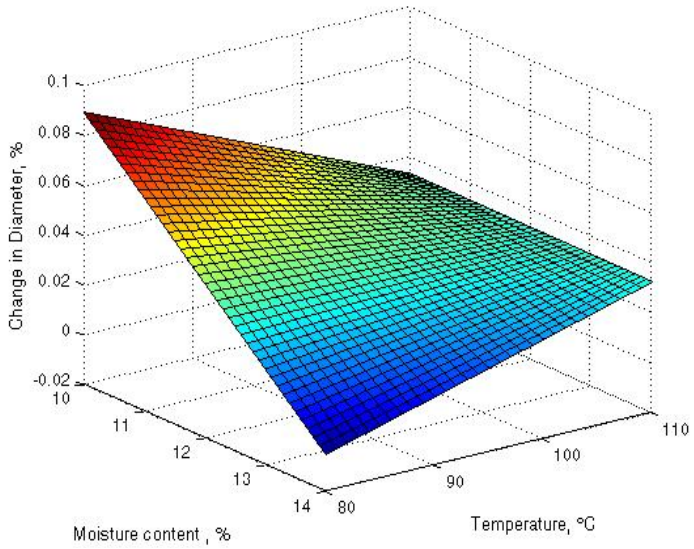

Figure 4.10 Lateral expansions of pellets showing interaction between moisture content and temperature

The only significant affect on the change in length to diameter ratio was an interaction between moisture content (X_1) and screen size (X_2) and the regression model equation is

$$\Delta l/d\ (\%) = 0.381744 - 0.02844\ X_1 - 0.02936\ X_2 + 0.002144\ X_1X_2 \quad (4.9)$$

Figure 4.11 shows that the change in length to diameter ratio increased with decreases in screen size and moisture content.

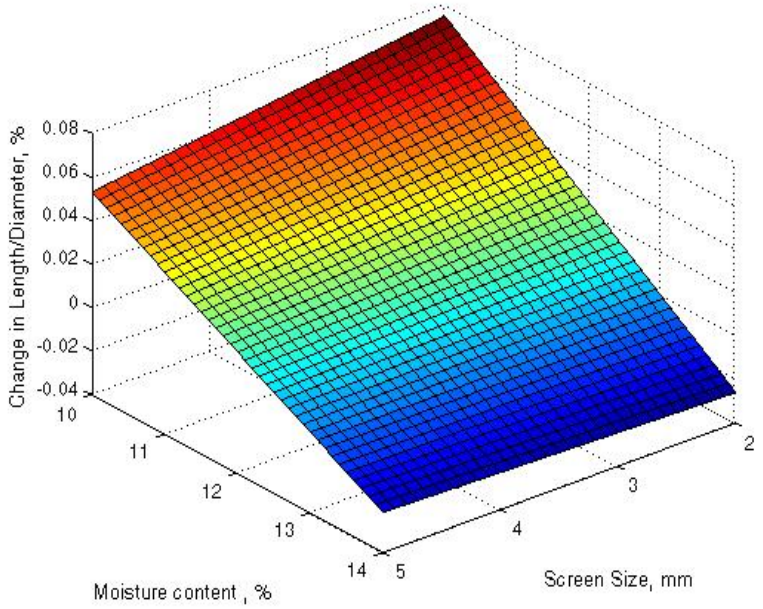

Figure 4.11 Change in length to diameter ratio showing interaction between screen size and moisture content

This trend is similar to that regarding the change in pellet length. This implies that the length to diameter ratio mainly depends on the change in pellet length and the estimate agrees with the experimental result shown in Table 4.9. The maximum change occurred at 11.5 moisture content and 3.2 mm screen size.

From the above results, it may be concluded that the dimensional stability of Terra Grain Fuels pellets was better than the Noramera pellets. This could be a result of the steam conditioning

used to form pellets from the Terra Grain Fuels DDGS. The pellets were more strongly bound together with steam conditioning and, therefore, were more dimensionally stable.

4.4.2 Bulk and particle density of pellets

4.4.2.1 Bulk and particle density of Noramera pellets

The bulk and particle densities and porosity of the pellets (mean values) are given below in Table 4.10. The result of the ANOVA performed on the data for these properties is given in Table 4.10.1. It shows that there is a significant affect on the bulk density due to screen size, moisture content and temperature but only with an interaction between screen size and temperature. However, for particle density, only screen size has statistical significance and there are no significant interactions among these factors.

Table 4.10 Bulk and particle density and porosity of Noramera pellets.

Parameters					
Screen Size (mm)	Moisture content (%), w.b.	Temperature (°C)	Bulk density (kg/m^3)	Particle density (kg/m^3)	Porosity (%)
3.2	15.5	100	691.27 (2.83)	1122.10 (1.40)	38.39 (0.33)
3.2	15.5	90	642.10 (6.17)	1117.16 (4.33)	42.52 (0.75)
3.2	14	100	703.41 (6.45)	1119.49 (2.36)	37.17 (0.45)
3.2	14	90	641.09 (3.41)	1112.05 (3.33)	42.35 (0.25)
4.8	15.5	100	663.39 (3.31)	1066.50 (6.22)	37.80 (0.11)
4.8	15.5	90	595.68 (2.91)	1033.26 (16.17)	42.32 (1.02)
4.8	14	100	677.82 (1.63)	1029.47 (10.80)	34.14 (0.69)
4.8	14	90	599.23 (2.93)	1032.00 (8.65)	41.92 (0.76)

* Values in parenthesis are standard error; n = 3

To examine the additional effects of the factors on the pellet bulk density, a linear regression analysis was performed. The regression model including parameters for moisture content (X_1) and screen size (X_2), and their interaction (X_1X_2) is

$$Bulk\ density = 727.7313+0.85533\ X_1-1.10667\ X_2-1.42667\ X_1X_2 \quad (4.10)$$

Figure 4.12, the plot of this regression equation, shows that the highest bulk density resulted from the smallest screen size and lowest moisture content. This agrees with experimental results at 3.2 mm screen size and 14% moisture content (w.b.). Earlier studies have shown the same trend. The bulk and particle densities of selected feedstock, namely poplar, pre-treated poplar, and wheat straw at two moisture levels (9 and 15% w.b.) using screen sizes of 0.8 and 3.2 mm were studied. Both, the bulk and particle densities of the feedstock decreased with an increase in moisture and screen size (Shaw 2008).

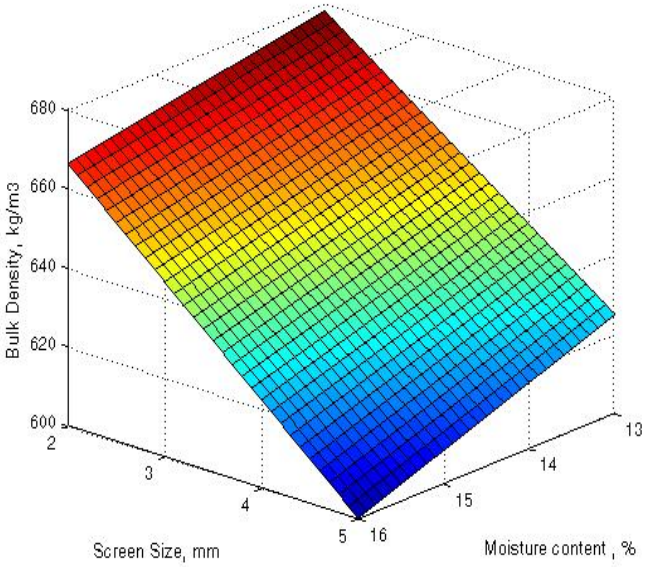

Figure 4.12 Bulk densities of Noramera pellets showing interaction between screen size and moisture content

The regression model for bulk density using screen size (X_2), temperature (X_3), and their interaction (X_2X_3) as parameters is

$$Bulk\ density = 541.5283-125.502\ X_2+2.092833\ X_3+1.087917\ X_2X_3 \quad (4.11)$$

Figure 4.13, the plot of this equation, shows that the bulk density increased with an increase in temperature and decrease in screen size. This estimate agrees with the experimental results, since the bulk density was highest for a screen size of 3.2 mm and a temperature of 100°C. This also agrees with earlier studies conducted on the bulk density of other biomass grinds. Flax shives, oat hulls and wheat straw pellets were studied and it was found that pellet densities of these biomass pellets increased with an increase in die temperature. Increasing the die temperature (60, 80 and 100°) successfully improved the pellet quality (Shaw and Tabil 2007). Preheating temperatures of 65 to 100°C were used for the study of various biomass products and helped in the manufacture of high quality products by pelleting, briquetting or cubing (Kaliyan and Morey 2008).

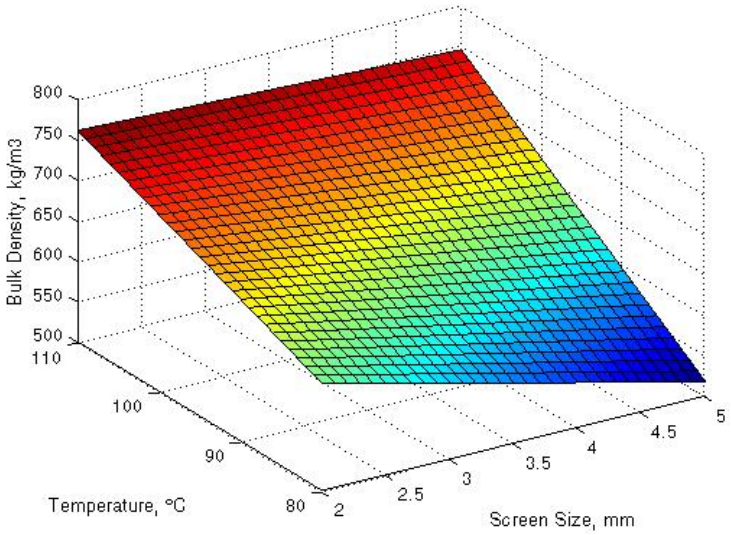

Figure 4.13 Bulk densities of Noramera pellets showing interaction between screen size and temperature

The final regression model produced for bulk density considered moisture content (X_1), temperature (X_3), and their interaction (X_1X_3). The equation is

$$Bulk\ density = -1011.79+71.27556\ X_1+18.26417\ X_3-0.80133\ X_1X_3 \quad (4.12)$$

Figure 4.14, the plot of this equation, shows that the highest bulk density occurs at the highest temperature and lowest moisture content, although moisture content does not seem to have a significant effect. This also agrees with the experimental results.

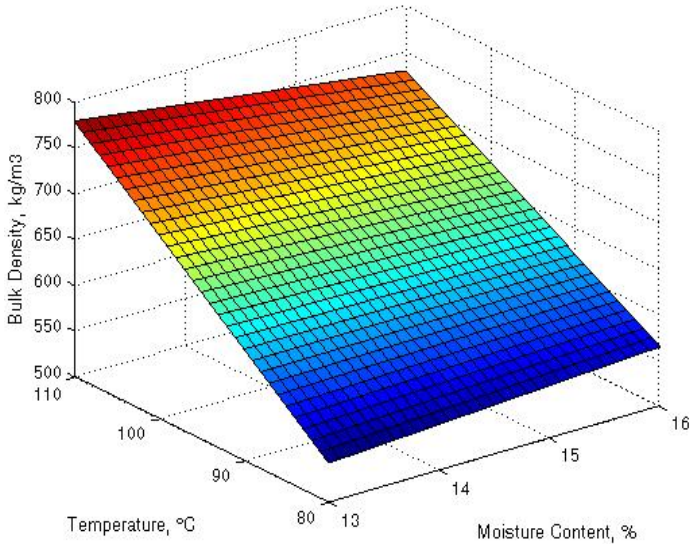

Figure 4.14 Bulk densities of Noramera pellets showing interaction between moisture content and temperature

The ANOVA results shown in Table 4.10.1 indicate that only screen size has a statistically significant effect on particle density.

Table 4.10.1 Mean square values of ANOVA of bulk and particle density; and porosity of Noramera Bioenergy Corp. pellets

Source	df	Bulk density	Particle density	Porosity
Screen size	1	7535.962*	35939.916*	6.772*
Moisture content	1	317.699*	793.730^{ns}	11.105*
Temperature	1	24918.948*	696.173^{ns}	175.052*
Screen size* Moisture content	1	17.579^{ns}	350.829^{ns}	2.645^{ns}
Screen size*Temperature	1	454.488*	125.858^{ns}	3.363^{ns}
Moisture content* Temperature	1	216.721^{ns}	415.002^{ns}	6.980*
Screen size*Moisture content* Temperature	1	1.938^{ns}	548.935^{ns}	1.811^{ns}
Error	16	48.754	198.389	1.141

*$p<0.05$, ns=not significant.

Therefore, to examine the effect of screen size with respect to moisture content and temperature, a regression analysis was performed. The regression model, including parameters for moisture content (X_1), screen size (X_2), and their interaction (X_1X_2) is

$$Particle\ density = 1535.352 - 17.8211\ X_1 - 142.362\ X_2 + 6.37222\ X_1X_2 \quad (4.13)$$

Figure 4.15 shows that with a decrease in screen size and increase in moisture content, the particle density increased. This estimate agrees with the experimental results, as the particle density was highest (1122 kg/m^3) for 15.5% (w.b.) moisture content and the 3.2 mm screen size.

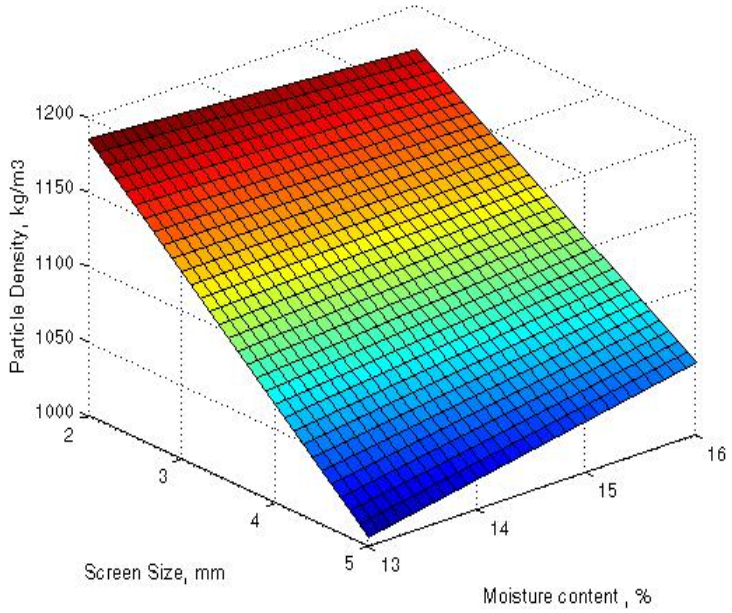

Figure 4.15 Particle densities of Noramera pellets showing interaction between moisture content and screen size

The regression model using screen size (X_2), temperature (X_3), and their interaction (X_2X_3) as parameters is

$$Particle\ density = 1387.71 - 102.759\ X_2 - 1.21283\ X_3 + 0.5725\ X_2X_3 \quad (4.14)$$

The graph in Figure 4.16 shows that the particle density increased with a decrease in screen size and an increase in temperature. This estimate agrees with the experimental results as the particle density was highest (1122 kg/m^3) for a screen size of 3.2mm and temperature of 100°C.

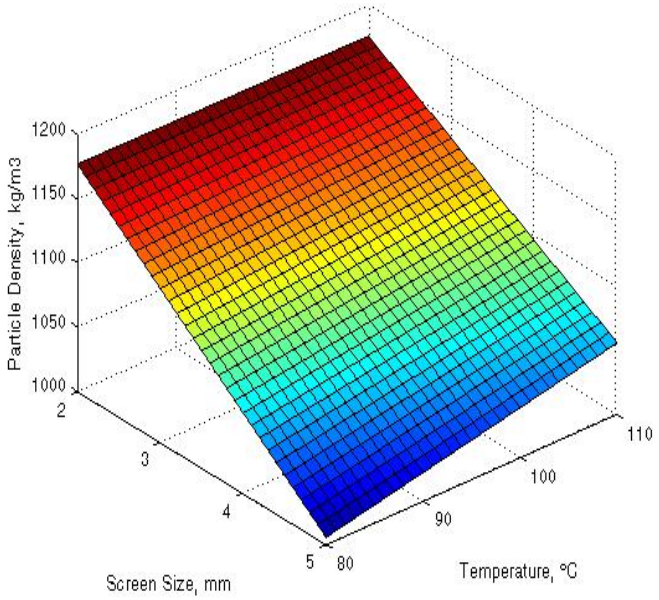

Figure 4.16 Particle densities of Noramera pellets showing interaction between screen size and temperature

4.4.2.2 Bulk and particle density of Terra Grain pellets

The bulk and particle densities and porosity (mean values) of the pellets are given below in Table 4.11. The ANOVA performed on the data (Table 4.11.1) shows that there is a significant effect on bulk density due to an interaction between moisture content and temperature and also, an interaction between all three factors. However, as for the Noramera pellets, particle density had only one significant factor, i.e., screen size.

Table 4.11 Bulk and particle density and porosity of Terra Grain pellets.

Parameters					
Screen Size (mm)	Moisture content (%), w.b.	Temperature (°C)	Bulk density (kg/m^3)	Particle density (kg/m^3)	Porosity (%)
3.2	13.09	100	541.63 (5.91)	1016.30 (6.74)	46.70 (0.52)
3.2	13.09	90	535.19 (7.52)	1011.63 (3.67)	47.09 (0.80)
3.2	11.5	100	611.25 (4.01)	1015.01 (3.73)	39.78 (0.18)
3.2	11.5	90	555.23 (3.66)	1008.83 (4.20)	44.96 (0.33)
4.8	13.09	100	566.93 (2.86)	988.53 (4.41)	42.65 (0.08)
4.8	13.09	90	525.53 (6.20)	991.86 (3.19)	47.01 (0.80)
4.8	11.5	100	610.65 (2.52)	996.12 (3.36)	38.69 (0.42)
4.8	11.5	90	567.95 (5.69)	993.13 (5.31)	42.81 (0.57)

* Values in parenthesis are standard error; n = 3

Table 4.11.1 Mean square values of ANOVA of bulk and particle density; and porosity of Terra Grain Fuels Ltd. pellets

Source	df	Bulk density	Particle density	Porosity
Screen size	1	288.982	2529.912*	20.404*
Moisture content	1	11587.857*	8.532^{ns}	111.062*
Temperature	1	8054.205*	41.475^{ns}	74.000*
Screen size* Moisture content	1	4.611^{ns}	62.953^{ns}	0.304^{ns}
Screen size*Temperature	1	175.500^{ns}	46.956^{ns}	3.164^{ns}
Moisture content* Temperature	1	971.045*	23.030^{ns}	7.757*
Screen size*Moisture content* Temperature	1	873.868*	8.724^{ns}	9.520*
Error	16	77.385	59.817	0.824

*$p<0.05$, ns=not significant.

To examine the additional effects of the factors on the pellet bulk density, a linear regression analysis was performed. The regression model that includes parameters for moisture content (X_1), screen size (X_2), and their interaction (X_1X_2) is

$$Bulk\ density = 920.666 - 30.3962\ X_1 - 4.13626\ X_2 + 0.689203\ X_1X_2 \quad (4.15)$$

Figure 4.17 shows that the highest bulk density resulted from the smallest screen size and lowest moisture content, which agrees with experimental results for 3.2 mm screen size, and 11.5% moisture content (w.b.).

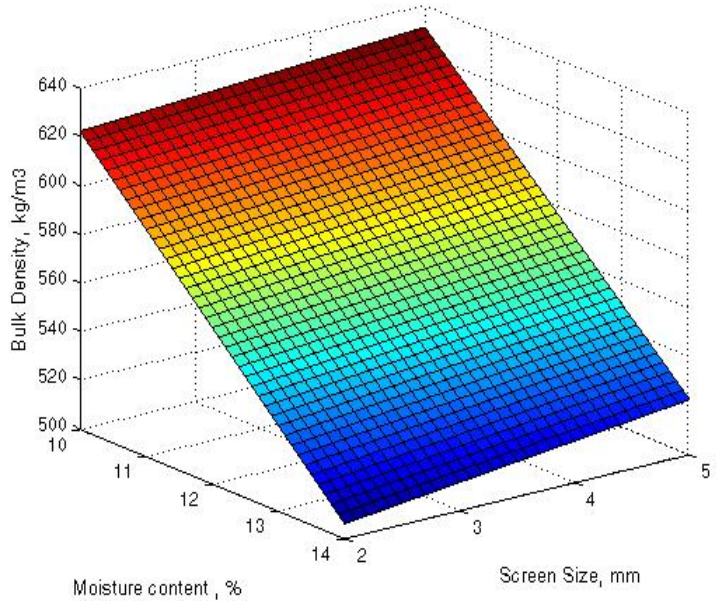

Figure 4.17 Bulk densities of Terra Grain pellets showing interaction between moisture content and screen size

The regression model that uses screen size (X_2), temperature (X_3), and their interaction (X_2X_3) as parameters is

$$Bulk\ density = 455.7767 - 59.8865\ X_2 + 0.959667\ X_3 + 0.676042\ X_2X_3 \quad (4.16)$$

Figure 4.18 show that higher bulk density was observed as the temperature increased and screen size decreased. The estimate agrees with the experimental results also as the highest bulk density (611.25 kg/m^3) occurred at 3.2 mm screen size and 100°C temperature.

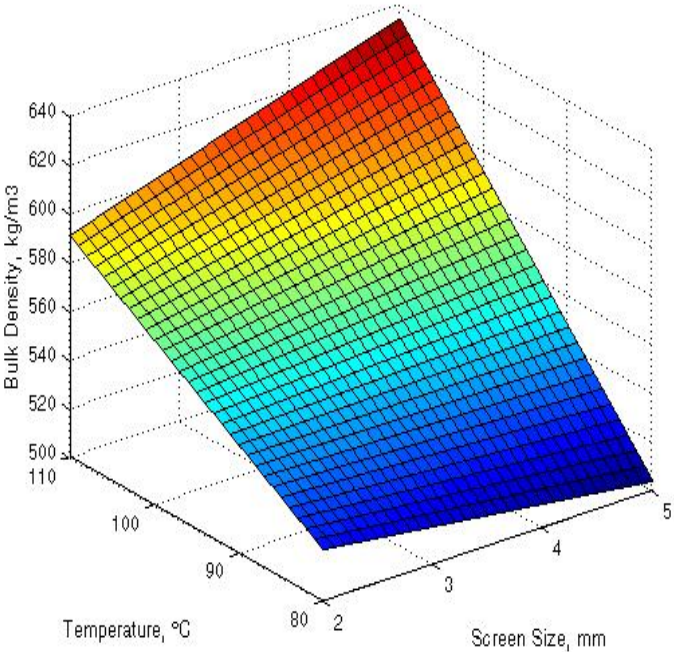

Figure 4.18 Bulk densities of Terra Grain pellets showing interaction between screen size and temperature

The last regression model performed for bulk density considered moisture content (X_1), temperature (X_3), and their interaction (X_1X_3). The equation is

$$Bulk\ density = -1313.03+124.3805\ X_1+23.33841\ X_3-1.60021\ X_1X_3 \quad (4.17)$$

Figure 4.19 show that the highest bulk density occurred for the highest temperature and lowest moisture content. This agrees with the experimental results as the highest bulk density (611.25 kg/m^3) was observed at 100°C and 11.5% (w.b.) moisture content. Preheating temperatures of 65 to 100°C were used on various biomass products and helped to manufacture high quality products by pelleting, briquetting or cubing (Kaliyan and Morey, 2008). A wide range of temperatures from 26 to 144 °C was studied on Norway spruce pellets and it was concluded that high temperature and low moisture content were the most important variables for increasing the compression strength and dry density of pellets (Rhen et al., 2005).

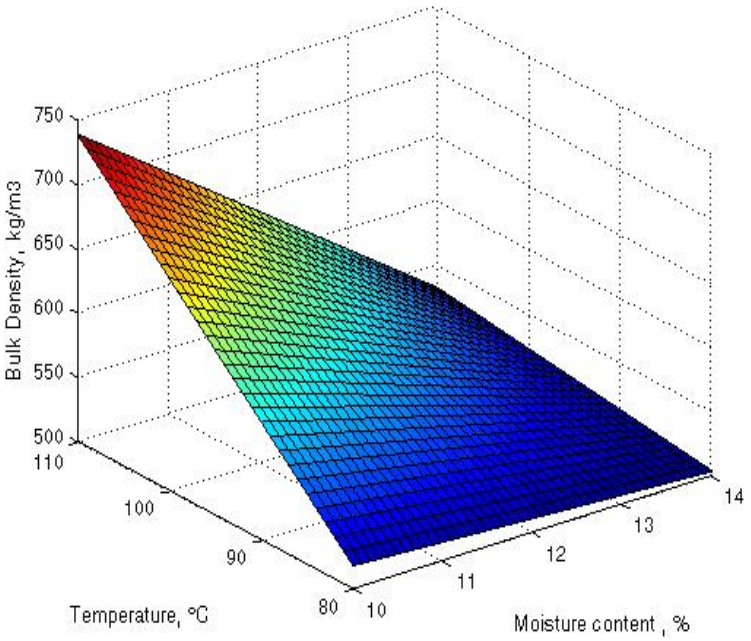

Figure 4.19 Bulk densities of Terra Grain pellets showing interaction between moisture content and temperature

The ANOVA results shown in Table 4.11.1 for particle density indicate that only screen size has a statistically significant effect. Therefore, to examine the effects of screen size with respect to moisture content and temperature, a regression analysis was performed. The regression model including parameters for moisture content (X_1), screen size (X_2), and their interaction (X_1X_2) is

$$Particle\ density = 937.9953+9.436059\ X_1+18.47555\ X_2-2.54651\ X_1X_2 \quad (4.18)$$

The graph of this equation, Figure 4.20, shows that particle density was highest for the smallest screen size and lowest moisture content. A previous investigation of wheat and barley straw grinds showed an increase in particle density of wheat straw grinds from 1030 to 1340 kg/m^3 when the geometric mean particle diameter was reduced, as a result of smaller screen size, from 0.64 to 0.28 mm (Mani et al 2006b).

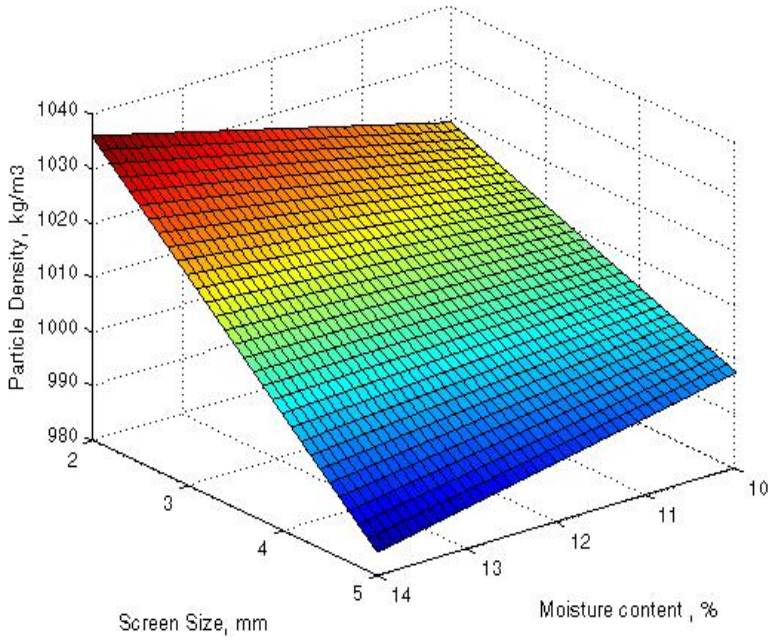

Figure 4.20 Particle densities of Terra Grain pellets showing interaction between screen size and moisture content

The regression model that uses screen size (X_2), temperature (X_3), and their interaction (X_2X_3) as parameters is

$$Particle\ density = 896.1533+20.38646\ X_2+1.66167\ X_3-0.34969\ X_2X_3 \quad (4.19)$$

Figure 4.21 shows the graph of this equation. The highest particle density was observed for the highest temperature and smallest screen size. This estimate agrees with the experimental results as the highest particle density of 1016.30 kg/m^3 was observed for a 3.2 mm screen size and temperature of 100°C. Earlier studies showed the same trend, too. The particle densities of wheat straw from screen sizes of 6.35 mm and 3.18 mm were 1085 kg/m^3 and 1210 kg/m^3, respectively (Mani et al. 2004b).

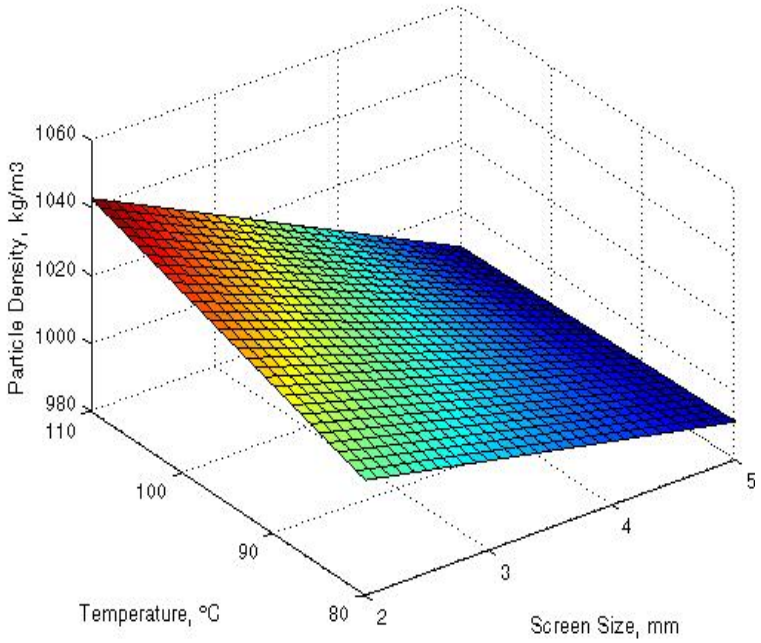

Figure 4.21 Particle densities of Terra Grain pellets showing interaction between screen size and temperature

In conclusion, the above results show that the highest bulk density occurred at a screen size of 3.2 mm and temperature of 100°C for sample pellets from both sources. The highest bulk density of the Noramera pellets was 703.41 kg/m^3 at a moisture content of 14% (w.b.). The highest bulk density of the Terra Grain pellets was 611.25 kg/m^3 at a moisture content of 11.5% (w.b.).

4.4.3 Hardness

Hardness is the measure of how resistant a material is to various kinds of permanent shape change when a force is applied**.**

4.4.3.1 Hardness of Noramera pellets

The mean experiment values of hardness are given in Table 4.12. The hardness test was analyzed using ANOVA. These results are shown in Table 4.12.1. The result shows that moisture content is the only property that has a significant affect on pellet hardness.

Table 4.12 Mean values of hardness of Noramera pellets.

Parameters			Hardness (N)
Screen size (mm)	Moisture content (%), w.b.	Temperature (°C)	
3.2	15.5	100	406.21 (13.53)
3.2	15.5	90	409.48 (20.77)
3.2	14	100	531.07 (9.16)
3.2	14	90	537.96 (11.61)
4.8	15.5	100	403.25 (10.65)
4.8	15.5	90	409.1 (14.64)
4.8	14	100	532.16 (17.44)
4.8	14	90	529.80 (5.72)

* Values in parenthesis are standard error; n = 10

Table 4.12.1 Analysis of variance of hardness of pellets from Noramera Bioenergy Corp.

Source	df	Mean Square
Screen size	1	135.382^{ns}
Moisture content	1	316244.903*
Temperature	1	233.006^{ns}
Screen size* Moisture content	1	17.011^{ns}
Screen size*Temperature	1	55.628^{ns}
Moisture content* Temperature	1	25.867^{ns}
Screen size*Moisture content* Temperature	1	173.844^{ns}
Error	72	1872.018

*$p<0.05$, ns=not significant.

To examine the effect of moisture content on hardness, a linear regression analysis was performed. The regression model including parameters for moisture content (X_1), screen size (X_2), and their interaction (X_1X_2) is

$$Hardness = 1758.237-86.9053\ X_1-12.9621\ X_2+0.768542\ X_1X_2 \quad (4.20)$$

Figure 4.22 is a plot of Equation 4.20. It shows that the pellet hardness increases with a decrease in moisture content and the screen size has no significant effect on the pellet hardness. An earlier study of switch grass pellets showed a decrease in hardness from 30.21 to 21.6 N with an increase in moisture content. The moisture disrupts particulate bonds, which leaves the pellets weak and susceptible to breakage (Colley et al. 2006). The results from this research follow a similar trend.

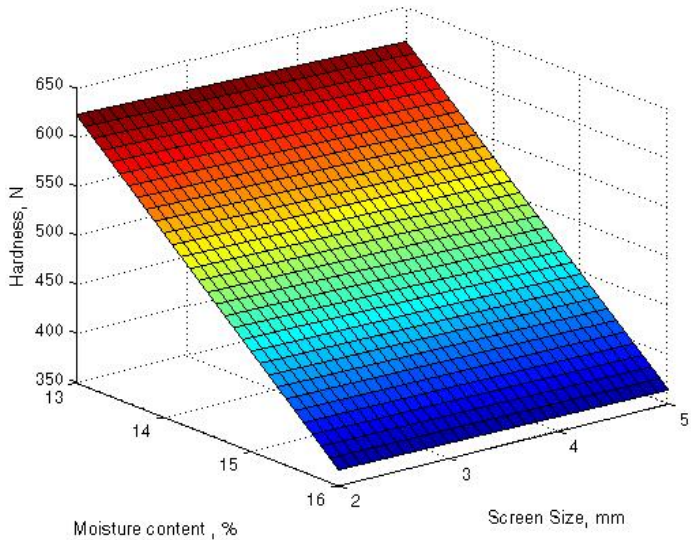

Figure 4.22 Effect of moisture content and screen size on Noramera pellet hardness

4.4.3.2 Hardness of Terra Grain pellets

The mean values of hardness for the Terra Grain pellets are given in Table 4.13. The results of the hardness test were analyzed using ANOVA. The ANOVA results are shown in Table 4.13.1. They show that only moisture content has a significant affect on pellet hardness. The regression model including parameters for moisture content (X_1), screen size (X_2), and their interaction (X_1X_2) is

$$Hardness = 2368.091 - 140.69\,X_1 + 34.5024\,X_2 - 2.84178\,X_1X_2 \quad (4.21)$$

Table 4.13 Mean values of hardness of Terra Grain Fuels pellets.

Parameters			Hardness (N)
Screen size (mm)	Moisture content (%), w.b.	Temperature (°C)	
3.2	13.09	100	518.35 (11.20)
3.2	13.09	90	517.32 (10.08)
3.2	11.5	100	770.39 (11.15)
3.2	11.5	90	741.59 (20.69)
4.8	13.09	100	511.81 (8.53)
4.8	13.09	90	515.23 (13.37)
4.8	11.5	100	762.75 (12.87)
4.8	11.5	90	755.06 (8.41)

* Values in parenthesis are standard error; n = 10

Table 4.13.1 Analysis of variance of hardness of pellets from Terra Grain Fuels Ltd.

Source	df	Mean Square
Screen size	1	9.793^{ns}
Moisture content	1	1169057.076*
Temperature	1	1453.598^{ns}
Screen size* Moisture content	1	261.328^{ns}
Screen size*Temperature	1	816.706^{ns}
Moisture content* Temperature	1	1889.471^{ns}
Screen size*Moisture content* Temperature	1	346.903^{ns}
Error	72	1584.243

*$p<0.05$, ns=not significant.

The regression equation 4.21 is plotted in Figure 4.23. It shows that the pellet hardness increases with decreases in moisture content and screen size; however the effect of screen size was not significant. The experimental results agree with this estimate. The highest value of pellet hardness was 770.39 N at a moisture content of 11.5% (w.b.) and screen size of 3.2 mm.

Therefore, the results show that the Terra grain pellets were harder than the Noramera pellets and in both cases the highest hardness value was a result of the lowest moisture content.

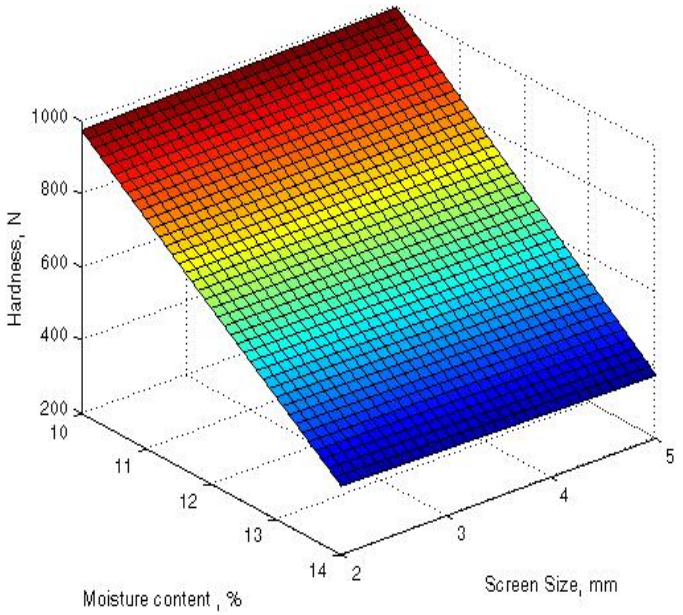

Figure 4.23 Effect of moisture content and screen size on Terra Grain pellet hardness

4.4.4 Durability of pellets

Durability of the densified biomass pellets is a very important factor as higher durability helps reduce breakage and crumbling of the products during transportation and handling.

4.4.4.1 Durability of Noramera pellets

The results of the durability tests on the Noramera pellets are shown in Table 4.14. The ANOVA results in Table 4.14.1 show that the screen size, moisture content and the interaction between screen size and temperature have significant effects on the durability.

Table 4.14 Mean values of durability of Noramera pellets.

Parameters			Durability (%)
Screen size (mm)	Moisture content (%), w.b.	Temperature (°C)	
3.2	15.5	100	93.15 (0.19)
3.2	15.5	90	91.85 (1.79)
3.2	14	100	83.55 (1.51)
3.2	14	90	86.41 (2.46)
4.8	15.5	100	86.99 (2.05)
4.8	15.5	90	80.73 (1.07)
4.8	14	100	80.61 (1.09)
4.8	14	90	77.75 (0.61)

* Values in parenthesis are standard error; n = 3

Table 4.14.1 Analysis of variance of durability of pellets from Noramera Bioenergy Corp.

Source	df	Mean Square
Screen size	1	312.99*
Moisture content	1	223.32*
Temperature	1	21.49^{ns}
Screen size* Moisture content	1	12.03^{ns}
Screen size*Temperature	1	42.85*
Moisture content* Temperature	1	21.49^{ns}
Screen size*Moisture content* Temperature	1	0.22^{ns}
Error	16	6.91

*p<0.05, ns=not significant.

To further verify the results of the ANOVA, regression analyses were performed. The regression model including parameters for moisture content (X_1) and screen size (X_2) is

$$Durability = 43.19772+4.066867\ X_1-4.51375\ X_2 \quad (4.22)$$

Figure 4.24 shows that the pellet durability was affected the most with decrease in screen size and increase in moisture content. This estimate agrees with the experimental results showing

that the highest durability occurred at a screen size of 3.2 mm and moisture content of 15.5% (w.b.). The result also agrees with previous research. In corn based DDGS, studies have shown that an increase in moisture content from 15 to 25 % (w.b.) resulted in a 28.2% increase in durability (Chevanan et al. 2008). In wheat DDGS pellets, a range of moisture contents from 11 to 16% (w.b.) was tested and the durability increased from 60% to 93% (Opoku et al. 2009). Another test on wheat based DDGS with moisture contents ranging from 5.10 to 11.80% (w.b) had a durability index ranging from 91.4 to 99.9 % (Tumuluru et al. 2010).

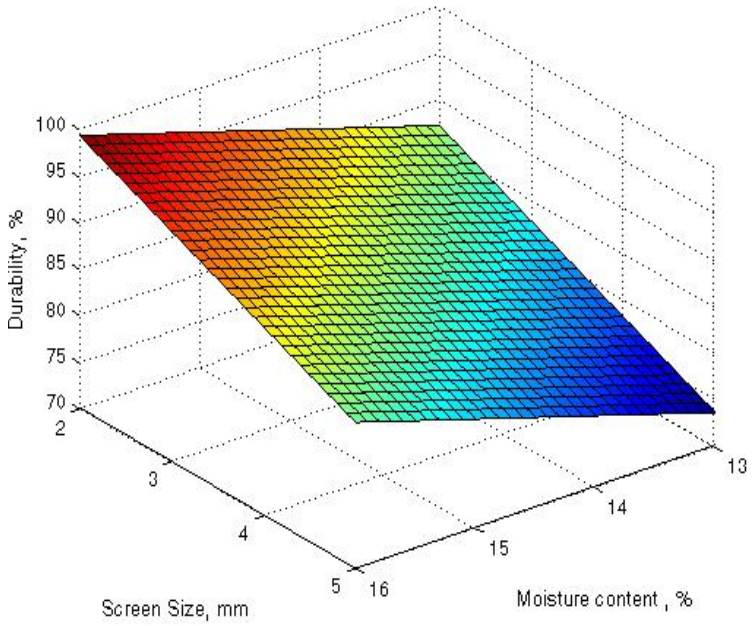

Figure 4.24 Effect of screen size and moisture content on durability of Noramera pellets

The regression model which uses, screen size (X_2), temperature (X_3), and their interaction (X_2X_3) as parameters is

$$Durability = 212.13-36.2438\ X_2-1.1468\ X_3+0.334\ X_2X_3 \quad (4.23)$$

Figure 4.25 is a plot of this equation. It shows that, essentially, two maximum changes of durability occurred as a result of these parameters. The combinations of the highest temperature and largest screen size; and lowest temperature and smallest screen size resulted in the largest increase in pellet durability.

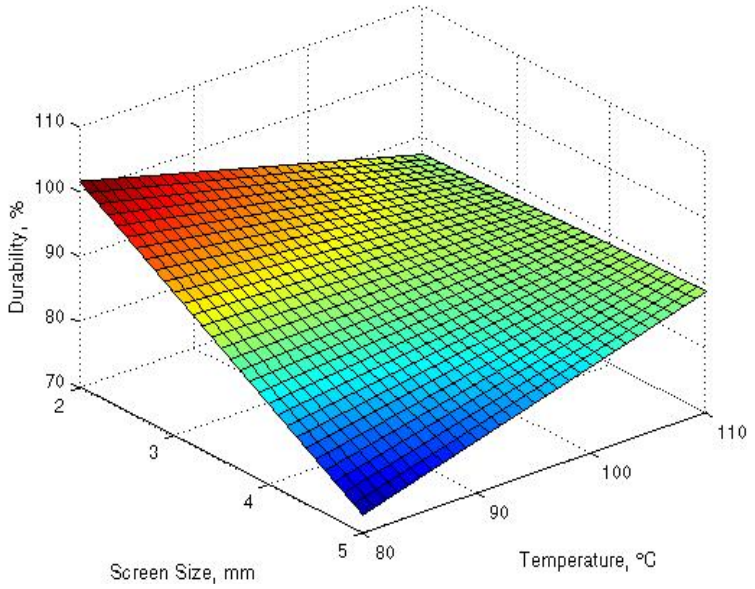

Figure 4.25 Effect of screen size and temperature on durability of Noramera pellets

4.4.4.1 Durability of Terra Grain pellets

The results of the durability test of the Terra Grain pellets are shown in Table 4.15. The ANOVA results are given in Table 4.15.1. These results show that there are significant affects on durability due to screen size, moisture content, temperature and interactions between screen size and moisture content, as well as, moisture content and temperature. The regression model including parameters for moisture content (X_1), screen size (X_2), and their interaction (X_1X_2) is

$$Durability = -81.1832+15.66216\,X_1 +29.67211\,X_2 -2.89957\,X_1X_2 \quad (4.24)$$

Table 4.15 Mean values of durability of Terra Grain pellets.

Parameters			Durability (%)
Screen size (mm)	Moisture content (%), w.b.	Temperature (°C)	
3.2	13.09	100	98.31 (2.64)
3.2	13.09	90	96.35 (3.46)
3.2	11.5	100	92.70 (1.20)
3.2	11.5	90	81.66 (3.45)
4.8	13.09	100	85.84 (1.84)
4.8	13.09	90	82.31 (1.18)
4.8	11.5	100	85.39 (1.43)
4.8	11.5	90	77.22 (2.87)

* Values in parenthesis are standard error; n = 3

Table 4.15.1 Analysis of variance of durability of pellets from Terra Grain Fuels Ltd.

Source	df	Mean Square
Screen size	1	549.031*
Moisture content	1	250.583*
Temperature	1	229.093*
Screen size* Moisture content	1	81.66*
Screen size*Temperature	1	0.637^{ns}
Moisture content* Temperature	1	70.487*
Screen size*Moisture content* Temperature	1	7.404^{ns}
Error	16	3.81

*$p<0.05$, ns=not significant.

Figure 4.26 shows a plot of Equation 4.24. In that plot, the durability increases as the screen size decreases and moisture content increases. This estimate agrees with the experimental results also as the highest durability occurred at a screen size of 3.2 mm and moisture content of 13.09% (w.b.).

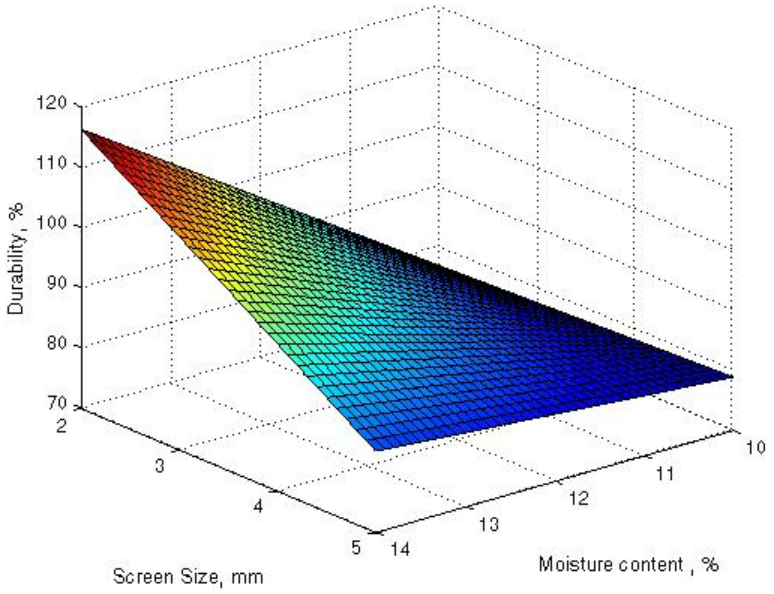

Figure 4.26 Affects of screen size and moisture content on durability of Terra Grain pellets

The final regression model created for durability considered moisture content (X_1), temperature (X_3), and their interaction (X_1X_3). The equation is

$$Durability = -524.896+45.03239\ X_1+5.920012\ X_3-0.43125\ X_1X_3 \quad (4.25)$$

The plot of this equation, Figure 4.27, shows that two maximum values that occurred for durability due to these parameters. Combinations of the highest temperature and lowest moisture content; and the lowest temperature and highest moisture content resulted in the largest values of pellet durability. Skoch et al. (1981) had similar results in their study of pellet durability. They found that pellet durability increased to 96.5% due to steam conditioning and fines were reduced during handling, transportation and feeding. They also determined that it was necessary to condition the grinds at 90°C or above so that pellet temperature would be high enough to promote better bonding of the particles during pelleting. Tabil (1996) found that increasing the conditioning temperature resulted in an increase in durability. Similarly, Hill and Pulkinen

(1988) using steam conditioning to raise the temperature of alfalfa mash from 60 to 104°C resulted in a 30 to 35% increase in alfalfa pellet durability.

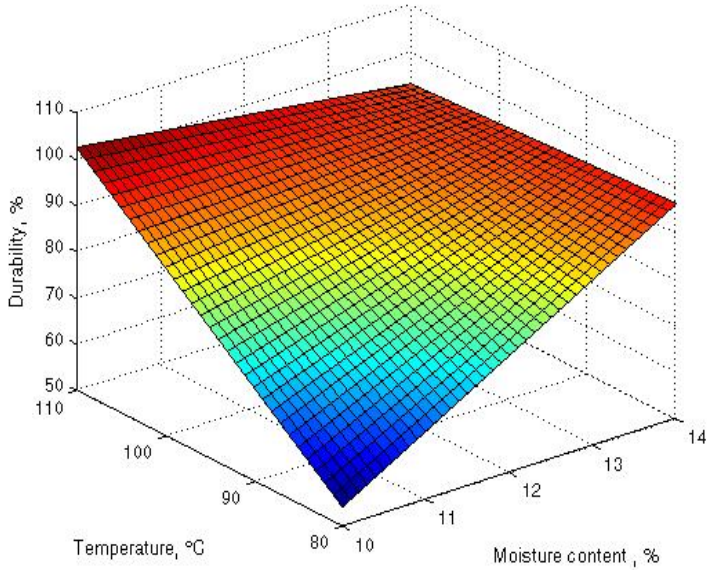

Figure 4.27 Effects of temperature and moisture content on durability of Terra Grain pellets

From the above results and discussion, we can conclude that the durability of Terra Grain pellets was higher than Noramera pellets because steam conditioning was incorporated during the production of the Terra Grain pellets. The steam conditioning likely activated the starch, lignin and protein present in the samples, which helped in better bonding of the particles. As a result, the pellets were more durable.

4.4.5 Moisture absorption

Moisture absorption is mainly studied because an increase in air temperature and relative humidity will cause an increase in the moisture absorption of pellets during storage, transportation and handling. Transportation during weather conditions that are rainy or have high humidity will affect the quality of pellets, making them more susceptible to breakage.

4.4.5.1 Moisture absorption of Noramera pellets

The results of the moisture absorption tests on Noramera pellets are given in Table 4.16. An ANOVA was performed and the results shown in Table 4.16.1 indicate that only moisture content had a significant affect. The regression model developed for the affect of moisture content (X_1) and temperature (X_3) on the moisture absorption of DDGS pellets is

$$\textit{Moisture absorption} = 618.7059 - 34.4864\, X_1 - 0.27751 X_3 \quad (4.26)$$

Table 4.16 Mean values of moisture absorption of Noramera pellets.

Parameters			Moisture absorption, w.b. (%)
Screen size (mm)	Moisture content (%), w.b.	Temperature (°C)	
3.2	15.5	100	55.72 (8.78)
3.2	15.5	90	58.56 (5.35)
3.2	14	100	111.77 (4.83)
3.2	14	90	112.69 (3.65)
4.8	15.5	100	56.48 (8.45)
4.8	15.5	90	60.46 (9.80)
4.8	14	100	105.16 (5.80)
4.8	14	90	108.51 (3.08)

* Values in parenthesis are standard error; n = 3

Table 4.16.1 Analysis of variance of moisture absorption of pellets from Noramera Bioenergy Corp.

Source	df	Mean Square
Screen size	1	24.788^{ns}
Moisture content	1	16055.687*
Temperature	1	46.206^{ns}
Screen size* Moisture content	1	67.845^{ns}
Screen size*Temperature	1	4.782^{ns}
Moisture content* Temperature	1	2.416^{ns}
Screen size*Moisture content* Temperature	1	0.630^{ns}
Error	16	132.387

*$p<0.05$, ns=not significant.

Figure 4.28 is a plot of Equation 4.26. It shows that the pellets with a lower moisture content absorbed more moisture. This estimate agrees with the experimental results also. Earlier studies done on Norway spruce pellets concluded that the highest moisture uptakes were found in samples with the lowest initial moisture content and vice versa (Rhen et al. 2005).

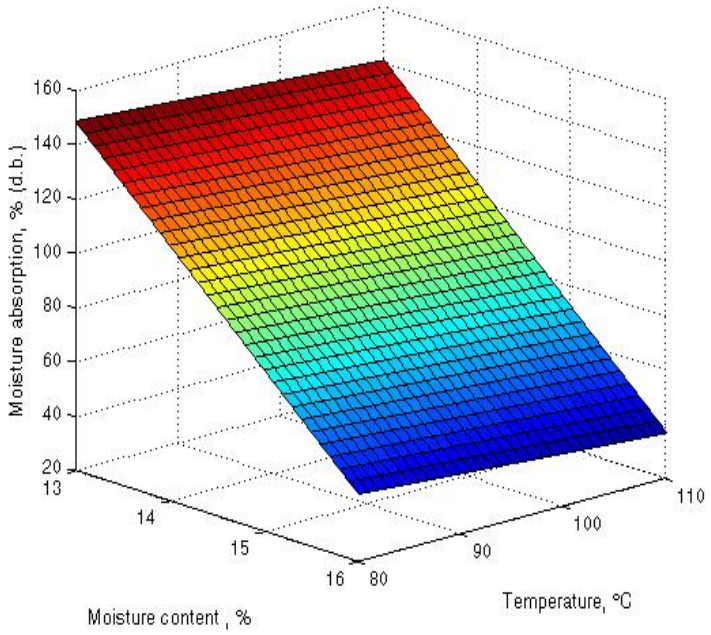

Figure 4.28 Effect of moisture content and temperature on moisture absorption of Noramera pellets

4.4.5.2 Moisture absorption of Terra Grain pellets

The results of moisture absorption tests performed on Terra Grain pellets are given in Table 4.17. The ANOVA results from the moisture absorption tests are shown in Table 4.17.1 and it indicates that moisture content was the only significant factor.

The regression model developed for the effect of moisture content (X_1) and temperature (X_3) on moisture absorption of Terra Grain DDGS pellets is

Moisture absorption = 371.8936-24.7086 X_1+0.103497 X_3 (4.27)

Table 4.17 Mean values of moisture absorption of Terra Grain pellets.

Parameters			Moisture absorption, w.b. (%)
Screen size (mm)	Moisture content (%), w.b.	Temperature (°C)	
3.2	13.09	100	56.53 (3.06)
3.2	13.09	90	60.57 (3.25)
3.2	11.5	100	101.01 (5.46)
3.2	11.5	90	95.94 (5.16)
4.8	13.09	100	59.46 (5.16)
4.8	13.09	90	56.60 (3.37)
4.8	11.5	100	96.81 (5.89)
4.8	11.5	90	96.55 (4.92)

* Values in parenthesis are standard error; n = 3

Table 4.17.1 Analysis of variance of moisture absorption of pellets from Terra Grain Fuels Ltd.

Source	df	Mean Square
Screen size	1	8.069^{ns}
Moisture content	1	9260.667*
Temperature	1	6.427^{ns}
Screen size* Moisture content	1	2.459^{ns}
Screen size*Temperature	1	1.647^{ns}
Moisture content* Temperature	1	15.858^{ns}
Screen size*Moisture content* Temperature	1	51.390^{ns}
Error	16	58.320

*$p<0.05$, ns=not significant.

The plot of equation 4.27 is Figure 4.29. It shows that the highest moisture absorption occurred in pellets with the lowest moisture content

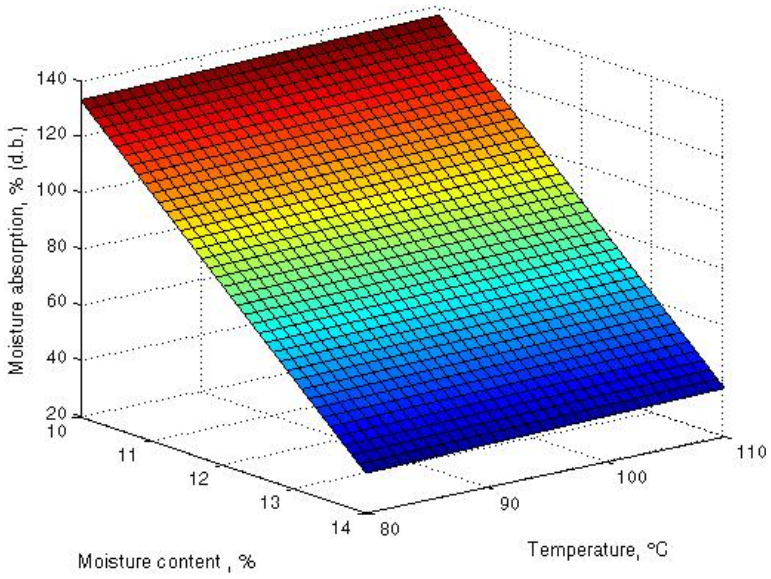

Figure 4.29 Effect of moisture content and temperature on moisture absorption of Terra Grain pellets

4.5 Ash content, Combustion energy and Emissions

4.5.1 Ash content

Ash is defined as the inorganic residue that remains after ignition of combustible substances and is determined by specific, prescribed methods (ASTM D121-09a). Ash content values are important because they help to determine the amount of ash that will be produced when the pellets are burned. The ash content was measured in triplicate for the DDGS produced from Noramera Bioenergy Corp. and Terra Grain Fuels Ltd. The DDGS were ground with 3.2 mm hammer mill screen size. The pellets used for the testing from Noramera Bioenergy were produced from DDGS with a moisture content of 15.5% (w.b.) using a heating temperature of 100°C whereas the DDGS from Terra Grain Fuels, had a moisture content of 13.09% (w.b.) and were produced with the same heating temperature.

The final moisture contents of the Noramera and Terra Grain pellets were 15.86% (w.b.) and 13.65% (w.b.), respectively. Table 4.18 shows the mean values of ash content produced from both samples. The results of one-way ANOVA are shown in Table 4.18.1.

Table 4.18 Ash content level in DDGS pellets.

DDGS pellet samples	Ash content (%)
Noramera Bioenergy Corp.	5.98 (0.11)
Terra Grain Fuels Ltd.	4.93 (0.21)

* Values in parenthesis are standard error; n = 3

Table 4.18.1 Analysis of varaiance of ash content of DDGS pellets.

Source of Variation	df	Mean square
Between Groups	3	11582.411*
Within Groups	20	8.034

* $p<0.05$

The experimental results show that Noramera Bioenergy pellets produced more ash than the Terra Grain pellets. This could be due to the activation of starch and lignin as a result of the steam conditioning used to produce pellets from the Terra grain samples. Less ash may also be produced due to the lower moisture content of the Terra Grain pellets.

4.5.2 Combustion energy

The combustion energies of pellets produced from both Noramera Bioenergy Corp. and Terra Grain Fuels Ltd. were measured in triplicate. The combustion energies of the pellets produced from Noramera and Terra Grain were 19.25 MJ/kg and 18.43 MJ/kg, respectively. The Noramera pellets had a higher combustion energy value than the Terra Grain pellets because of the high percentage of dry matter, ash content and lignin present in their samples.

4.5.3 Emissions

Emission tests were performed on the Noramera and Terra Grain DDGS pellets. These were compared to the commercial wood pellets, shown in Figure 4.30. The DDGS pellets were produced with DDGS ground with a 3.2 mm hammer mill screen size and moisture contents of the pellets were 15.5% (w.b.) and 13.09% (w.b.) for the Noramera and Terra Grain pellets, respectively.

Figure 4.30 Commercial wood pellets

Table 4.19 shows the results of the emission tests performed on the different pellets. The ANOVA results in Table 4.19.1 show that there was no significant difference in the emissions between the DDGS pellets. The emissions of the DDGS pellets from both samples were similar but varied from the commercial wood pellets. The wood pellets had lower levels of nitrous oxide and nitrogen but a higher level of carbon dioxide.

Table 4.19 Emission values of DDGS and wood pellets.

Pellets	Emissions				
	Nitrous oxide (ppm)	Methane (ppm)	Carbon dioxide (ppt)	Nitrogen (ppt)	Oxygen (ppt)
Noramera	6.04 (0.41)	1.42 (0.07)	24.88 (1.74)	796.25 (0.54)	189.47 (1.91)
Terra Grain	7.30 (0.71)	1.52 (0.10)	26.17 (2.35)	797.09 (0.69)	188.12 (2.60)
Commercial wood	0.50 (0.01)	1.52 (0.24)	28.11 (1.72)	793.59 (0.23)	188.55 (1.77)

*Value in parenthesis is standard error, n=7.
ppm = parts per million.
ppt = parts per thousand.

Table 4.19.1 Mean squares from analysis of variance of emissions of DDGS and wood pellets from combustion.

Source	df	Nitrous oxide (ppm)	Methane (ppm)	Carbon dioxide (ppt)	Nitrogen (ppt)	Oxygen (ppt)
Pellets	20	10.603*	0.155*	26.062*	4.067*	28.788*
Error	62	0.56	0.044	2.68	0.345	1.743

*$p<0.05$

4.6 Summary

The initial moisture contents of the coarse/unground samples from Noramera and Terra Grain were 12.5% and 13.75% (w.b.), respectively. Grinding with a hammer mill caused the moisture content of the materials to decrease. This indicates that heat is generated during grinding with a small screen.

For the coarse/unground DDGS Noramera, the geometric mean diameters were 2.496 mm and 1.461 mm for Noramera and Terra Grain samples, respectively. These corresponded with geometric standard deviations of 1.57 and 1.36 mm, respectively. For Noramera DDGS,

hammer mill screens of 4.8 and 3.2 mm resulted in geometric mean diameters of 0.783 and 0.726 mm, respectively and the Terra Grain samples had values of 0.516 and 0.495 mm, respectively. Grinds produced with the 3.2 mm screen size had standard deviations of 0.62 and 0.39 mm, respectively. These were lower than the standard deviations from the 4.8 mm screen. The lower standard deviations indicate that the material became more uniform in size.

When the particle size distribution was determined for the Noramera samples, most of the material was retained on the sieve with openings of 1.19 mm for both the screen sizes. Overall, the particles on 2.00 to 0.42 mm screens had large size distributions. The amount of material retained on the sieves from 0.21 to 0.04 mm was very low because the majority of the samples were retained on the larger screen sizes. For Terra Grain samples, most of the material was retained on the sieve with openings of 0.42 mm. Large size distributions were seen on the sieves with opening sizes of 1.19 to 0.25 mm. As occurred for the Noramera samples, very small amounts of material were retained on the sieves with opening sizes of 0.18 to 0.04 mm. The initial bulk and particle densities of the coarse DDGS from Noramera Bioenergy Corp. and Terra Grain Fuels Ltd. were determined to be, on average, 658.8 and 1135.0 kg/m^3; and 451.5 and 809.4 kg/m^3, respectively.

The coefficient of friction was similar in both samples. However; cohesion was higher (8.534 kPa) in Noramera samples than in Terra Grain samples (7.402 kPa).

Noramera samples had a higher percentage of lignin (10.5%) in comparison to the Terra Grain samples (4.37%). As a result, steam conditioning was required to form pellets with the Terra Grain samples. The protein in the Noramera samples was higher and helped, to increase the durability of the pellets. The combustion energy of the Noramera samples was 19.45 MJ/kg at a

moisture content of 8.6% (w.b.) whereas the combustion energy of Terra Grain samples was 18.54 MJ/kg at 12.31% (w.b.) moisture content.

There was a significant affect from grinding on the durability of the pellets. The durability of the pellets ranged from 80 to 92%. The use of 3.2 mm screen resulted in the highest durability (92.25%) of pellets. They were produced from Terra Grain Fuels samples.

The effects of two levels of moisture content, screen size and temperature on various pellet properties were investigated. The pellets produced from Noramera had a larger change in length for a moisture content of 14% than for 15.5% (w.b.). Pellets formed at the higher moisture content did not absorb as much moisture, and therefore, the pellet length did not increase as much. The statistical analysis (ANOVA) showed that the change in diameter occurred mainly due to the interaction between screen size and temperature; and between moisture content and temperature.

The pellets produced from Terra Grain Fuels material showed that the change in pellet length increased with low moisture content and small screen size, although the affect of screen size was not significant. The largest change in pellet length occurred for a moisture content of 11.5% (w.b.) and screen size of 3.2 mm. From the experimental results, the dimensional stability of Terra Grain pellets was higher in comparison to the Noramera pellets. This may be due to the steam conditioning of the Terra Grain Fuels pellets.

The ANOVA performed for the bulk and particle densities of Noramera samples showed that there was a significant affect on bulk density in regards to screen size, moisture content and temperature but only with interaction between screen size and temperature. However, for particle density only screen size has statistical significance. The highest bulk density resulted

from the smallest screen size and lowest moisture content. The particle density was lower for the smaller screen size and higher moisture content. This estimate agrees with the experimental results. The particle density was highest (1122 kg/m^3) at 15.5% (w.b.) moisture content and 3.2 mm screen size.

The ANOVA performed for the bulk and particle densities of Terra Grain Fuel samples, shows that there is significant effect on bulk density with regard to moisture content, temperature, their interaction and the interaction of all three factors. However, screen size was the only significant factor for particle density. The highest bulk density resulted from the smallest screen size and lowest moisture content. The highest particle density occurred for the highest temperature and smallest screen size. This estimate agrees with the experimental results. The highest particle density, 1016.30 kg/m^3, was observed for a screen size of 3.2 mm screen size and temperature of 100°C.

The only significant factor on pellet hardness was moisture content. For the Noramera pellets, hardness values varied from 402-538 N and it was highest at a moisture content of 14% (w.b.). In the Terra Grain pellets, the highest hardness was 770.39 N at 11.5% (w.b.) moisture content and the values ranged from 510-770 N.

In durability tests of the pellets, the only significant factor was the interaction between screen size, moisture content and temperature. In moisture absorption tests, the only significant factor was moisture content for both samples. This shows that pellets with lower moisture contents absorb more moisture.

Noramera Bioenergy pellets produced more ash (5.98%) compared to Terra Grain Fuels pellets (4.93%). This may be due to the steam conditioning or the lower moisture content of the Terra Grain Fuels samples.

Pellets produced from Noramera DDGS had a combustion energy of 19.25 MJ/kg while the pellets produced from Terra Grain DDGS had a combustion energy of 18.43 MJ/kg. The higher value of the Noramera pellets is because of the high percentage of dry matter, ash and lignin present in their samples.

There was a significant difference in emission values between DDGS pellets and commercial wood pellets. Wood pellets had a lower level of nitrous oxide (0.50 ppm) in comparison to 6.04 and 7.30 ppm produced by the DDGS pellets. The carbon dioxide level was higher in the wood pellets, i.e., 28.11 ppt compared to 24.88 and 26.17 ppt in DDGS. The methane, nitrogen and oxygen values were similar for all of the pellets.

5. CONCLUSION

There has been an increase in the cost and use of non-renewable fossil fuels in the past decade and it has been a hot topic of discussion worldwide. The problem could be solved if energy dependency is reduced or alternative methods of energy production are introduced. With advances in automobile technology, etc., energy dependency would decrease. However, that seems to be far from reality. So, to develop alternative sources of energy, researchers have been working on renewable sources of energy like wind, solar, hydro, etc. Biomass is a source of renewable energy, which if commercialized, could meet human energy requirements. Biomass includes wood wastes, agricultural wastes, bagasse, industrial residues, alcohol fuels, sawdust, bio-solids, grass waste from food processing, crop wastes, grasses, legumes, biological wastes, etc. The key issue with biomass is the handling as they are costly to transport and store due to their low bulk density. To overcome this problem, researchers came up with the idea of compacting the biomass into cubes, pellets and briquettes. This results in an increase in the bulk density of the final product.

The densification of different biomass is affected by the raw material properties and the processing parameters. The material properties of concern include: particle size, particle size distribution, bulk density, particle density, chemical composition, and moisture content. The pertinent properties of the processed pellets include: stability, density, hardness, durability and moisture absorption. These were studied, at length in this research, relative to the parameters: screen size, moisture content and temperature.

In this investigation, DDGS from two fuel ethanol plants were obtained and used to manufacture bio-fuel pellets using a pilot scale-pellet mill, with and without steam conditioning.

The first objective of the research was to characterize the DDGS from both plants on the basis of physical and chemical properties. From this work, these conclusions can be drawn:

1. The moisture content of DDGS from both sources was similar. Noramera Bioenergy samples had initial moisture content of 12.5% (w.b.) in comparison to 13.75% (w.b) for the Terra Grain Fuels samples.
2. The DDGS were ground using hammer mill screen sizes of 3.2 and 4.8 mm.
    a) The moisture content reduced with a decrease in screen size in both the samples. The moisture contents were 11.6% (w.b.) and 11.5 % (w.b) for the Noramera and Terra Grain DDGS, respectively.
    b) The DDGS ground with a 3.2 mm screen size had particles with a smaller mean geometric diameter than those ground with a 4.8 mm screen size.
3. There was an increase in bulk and particle density with decreased hammer mill screen size. The bulk and particle densities increased by 12 and 13%, respectively when the 3.2 mm screen size was used.
4. The coefficient of friction was similar for both samples. However, cohesion was greater in the Noramera Bioenergy samples (i.e., 8.534 kPa) than in the Terra Grain Fuels samples (i.e., 7.402 kPa).
5. Noramera Bioenergy samples had a higher percentage of lignin (i.e., 10.5%) compared to the Terra Grain Fuels samples (i.e., 4.37%). Crude fibre was higher (i.e., 7.33%) in the Terra Grain Fuels samples than in the Noramera Bioenergy samples (i.e., 4.98%).
6. The cellulose quantity was similar in both samples at approximately 11%. Hemi-cellulose was higher in Terra Grain Fuels samples (i.e., 27.45%) than in Noramera Bioenergy samples (i.e., 21.04%). The fat content was higher in the Terra Grain Fuels sample (i.e.,

6.37%). The moisture contents of the DDGS samples in this study were 8.6% and 12.31% (w.b.) for Noramera Bioenergy and Terra Grain Fuels samples, respectively.

7. The combustion energy of the Noramera Bioenergy samples was 19.45 MJ/kg at moisture content of 8.6% (w.b.) whereas the combustion energy of Terra Grain Fuels samples was 18.54 MJ/kg at 12.31% (w.b.) moisture content.

The second objective was to examine the pellets produced from the DDGS described above on the basis of particle size, bulk density, particle density, moisture absorption, hardness and durability with regard to the parameters of screen size, moisture content and temperature of production.

1. A reduction in screen size resulted in pellets with higher durability. The 3.2 mm screen size produced more durable pellets. The mean values of durability are 88.74% and 92.25% for Noramera Bioenergy and Terra Grain Fuels samples, respectively.
2. In terms of dimensional stability, the Noramera pellets with the smallest change in length and diameter were produced from DDGS ground with a 4.8 mm screen size, and having a moisture content of 15.5% (w.b.) and production temperature of 100°C. The lowest change in the ratio of length/diameter was not obvious. However, it seemed to follow the same trend as the change in pellet length. The Terra Grain pellets with the lowest change in length were produced with DDGS ground with a 4.8 mm screen size, and having a moisture content of 13.09% (w.b.) and production temperature 90°C. There were no Terra Grain pellets, which clearly had the lowest change in diameter or length/diameter ratio.
3. The highest bulk density of Noramera pellets (i.e., 703.41 kg/m^3) were produced from DDGS ground with a 3.2 mm screen size, and having a 14% (w.b.) moisture content and

100°C temperature. The highest bulk density for Terra Grain Fuels pellets (i.e., 611.25 kg/m^3) were produced at 11.5 % (w.b.) moisture content.

4. The highest particle density for Noramera pellets was 1122.10 kg/m^3 with a 3.2 mm screen size, 15.5% (w.b.) moisture content and 100°C temperature; whereas, for Terra grain pellets, the highest was 1016.30 kg/m^3 with a 3.2 mm screen size, 13.09% (w.b.) moisture content and 100°C temperature.
5. Moisture content was the only statistically significant factor with regard to pellet hardness. The highest hardness of Noramera pellets was 537.96 N at 14% (w.b.) moisture content and for Terra Grain pellets; it was 770.39 N at 11.5% (w.b.) moisture content.
6. The durability was affected by moisture content, screen size and temperature. The highest durability of Noramera pellets was 93.15% at 3.2 mm screen size, 15.5% (w.b.) moisture content and 100°C temperature. The highest durability of Terra Grain Fuels pellets was 98.31% at 3.2 mm screen size, 13.09% (w.b.) moisture content and 100°C temperature.
7. Moisture content was the only significant factor with regard to moisture absorption, too. In Noramera Bioenergy pellets, the lowest moisture absorption was observed at 15.5% (w.b.) moisture content whereas in Terra Grain Fuels pellets, it occurred at 13.09% (w.b.) moisture content.

The third and final objective of the study was to test the pellets for heat of combustion (i.e., gross energy), ash content, and fuel gas emissions.

1. The ash content of the Noramera pellets (i.e., 5.98%) was higher than the Terra Grain Fuels pellets (i.e., 4.93%). This is due to the lower moisture content of the Terra Grain Fuels pellets.

2. Pellets produced from Noramera Bioenergy DDGS had a combustion energy of 19.25 MJ/kg while the pellets produced from Terra Grain Fuels DDGS had a combustion energy of 18.43 MJ/kg. The combustion energy of the Noramera pellets was higher because of the higher percentage of ash present in them.
3. There was no significant difference in emission measurements from either sample except for the percentage of nitrous oxide and methane. Nitrous oxide was higher from Terra Grain Fuels pellets (i.e., 7.30 ppm) compared to Noramera Bioenergy pellets (i.e., 6.04 ppm). Methane was also higher from Terra Grain Fuels samples (i.e., 1.52 ppm). There was a significant difference in When the emission values of DDGS pellets were compared to commercial wood pellets, The DDGS pellets had significantly lower levels of carbon dioxide (i.e., 24.88 and 26.17 ppt) compared to the wood pellets (i.e., 28.11 ppt).

Finally, from this research, it can be concluded that the pellets produced from distillers' grains with solubles may be commercially produced and used for burning in furnaces to generate heat. The gases produced from burning DDGS are non-toxic. So, the health of the farmers and their livestock will not be affected.

6. RECOMMENDATIONS FOR FUTURE WORK

The following list of suggestions was compiled for future research studies:

1. Grinding biomass with other screen sizes may be considered for increasing or decreasing the bulk density. However, a 3.2 mm screen size seems to be adequate for creating fuel pellets. A smaller screen size would require more energy, thereby increasing the cost of production.
2. Moisture contents of 11 to 16% seem to be ideal for the production of pellets with or without steam conditioning. However, moisture contents lower than this, e.g., 8% and 10%, could be used to make pellets depending upon the quality of the biomass. However, if the biomass is too dry, water addition may make it too wet for production of pellets by causing the die and roller assembly in the pellet mill to clog.
3. The temperature could be varied from 75 to 100°C. However, increasing the temperature above 100°C is not recommended. More research needs to be performed regarding lower levels of temperature and the optimum temperature for pelleting needs to be better established.
4. There is a need to determine the optimum pressure required to compact the biomass as the materials are going into the die. Pressures between 50 and 150 MPa have been previously used for compaction of other biomass materials.
5. Binders could be used to enhance the quality of the pellets. Other biomass materials, such as saw dust, canola meal, etc., could also be added in the production of the DDGS pellets.

6. Advancements in technological equipment are required to enable commercial levels of pellet production. Other processing parameters, such as pressure, die geometry, speed, time etc., could be considered for production of pellets.
7. The energies required to compact and grind the biomass could be studied, in detail.
8. Emission measurements of other greenhouse gases could be studied to determine their effects on the environment.

REFERENCES

AOAC International. 2000. Official Method 923.03. Ash of flour. In *Official Methods of Analysis of AOAC International.* 17th ed. Gaithersburg, MD, USA: Association of Official Analytic Chemists.

AOAC International. 2002. Official Method 984.13. Protein (crude) in animal feed and pet food. In *Official Methods of Analysis of AOAC International.* 17th ed. Gaithersburg, MD, USA: Association of Official Analytic Chemists.

AOAC International. 2006. Official Method 920.39. Fat (crude) or ether extract in animal feed. In *Official Methods of Analysis of AOAC International.* 18th ed. Gaithersburg, MD, USA: Association of Official Analytic Chemists.

ANKOM Technology, 2005. ANKOM Technology Method. http://www.ankom.com/media/documents//ManualA200_REVB06108.pdf (2010/08/20)

ASABE Standards, Dec 1991 (R2007). ASAE S269.4. Cubes, pellets, crumbles Definitions and methods for determining density, durability and moisture content. *In ASABE Standards 2007.* St. Joseph, MI: ASABE.

ASABE Standards 2008. ANSI/ASAE S319.4 FEB 2008. Method of determining and expressing fineness of feed materials by sieving.

ASABE Standards, Dec 2008 (R 2008). ASAE S368.4. Compression test of food materials of convex shape. *In ASABE Standards 2009.* St. Joseph, MI: ASABE.

ASAE Standards. 2005. S358.2: Moisture measurement-forages. *In ASAE Standards 2005.* 52nd ed. St. Joseph, MI: ASAE.

ASTM Standard E 711-87. ASTM 2004. Standard test method for gross calorific value of refuse-derived fuel by the bomb calorimeter.

ASTM Standard D1102-84. ASTM (Reapproved 2007). Standard test method for ash in wood.

Adapa, P. K., L. G. Tabil, G. J. Schoenau and S. Sokhansanj. 2004. Pelleting characteristics of fractionated sun-cured and dehydrated alfalfa grinds. *Applied Engineering in Agriculture* 20(6):813-820.

Adapa, P., L. Tabil and G. Schoenau. 2009. Compaction characteristics of barley, canola, oat and wheat straw. *Biosystems Engineering.* 104:335-344.

Ag Fuel & Feed. 2010. http://www.agfuelandfeed.com/ (2010/12/02).

Bhadra, R., K. Muthukumarappan and Kurt. A. Rosentrater. 2007. Characterization of chemical and physical properties of distillers' dried grains with solubles (DDGS) for value added uses. ASABE Paper No. 077009. Minneapolis, MN: ASABE.

Boman, C., A. Nordin., and L. Thaning. 2003. Effects of increased biomass pellet combustion on ambient air quality in residential areas - a parametric dispersion modeling study. *Biomass and Bioenergy* 24(6):465–74.

Boman, C., A. Nordin, D. Boström and M. Öhman. 2004. Characterization of inorganic particulate matter from residential combustion of pelletized biomass fuels. *Energy & Fuels* 18: 338-348.

British Standard BS 2955. 1991. Glossary of Terms Relating to Particle Technology, British Standards Institution, London.

Canadian Renewable Fuels Association. 2010. http://www.greenfuels.org/ (2010/06/06).

Coates, W. 2000. Using cotton plant residue to produce briquettes. *Biomass and Bioenergy* 18:201-208.

R. C. Christiansen. 2009. Supply and Demand of DDGS. *Ethanol Producer Magazine*: March issue.

R. C. Christiansen. 2009. Perfecting the DDGS Pellet. *Ethanol Producer Magazine*: June issue.

Chevanan, N., K. A. Rosentrater and K. Muthukumarappan. 2008. Effects of DDGS, moisture content, and screw speed on physical properties of extrudates in single-screw extrusion. *Cereal Chemistry.* 85(2): 132-139.

Chung, J. H., and L. R. Verma. 1989. Determination of friction coefficients of beans and peanuts. *Transactions of the ASAE.* 32(2):745-750.

Colley, Z., Fasina,O.O., Bransby, D. and Y.Y. Lee. 2006. Moisture effect on the physical characheristics of switchgrass pellets. *Transactions of the ASABE*
49(6): 1845-1851.

Demirbas, A. 2004. Combustion characteristics of different biomass fuels. *Progres in Energy and Combustion Science*, Elsevier 30: 219-230.

Distillers grains technology Council. 2010. http://www.distillersgrains.org/grains/availability.html (2010/06/06).

Dobie, J.B. 1959. Engineering appraisal of hay pelleting. *Agricultural Engineering* 40(2):76-77,92.

The Fish Site. 2010. http://www.thefishsite.com/fishnews/13196/success-for-japanese-aquaculture-and-ddgs-trial (2010/29/12).

Heffner, L.E. and H.B.Pfost. 1973. Gelatinization during pelleting. *Feedstuff* 45(23):33.Hoekman, S. K. 2009. Biofuels in the U.S.- Challenges and opportunities. *Renewable Energy* 34:14-22.

Ganesan, V., K. Muthukumarappan, and K. A. Rosentrater. 2006. Effects of flow agents addition on the physical properties of DDGS with varying moisture content and soluble levels. ASABE Paper No. 066076.

Government of Alberta. 2010.
http://www1.agric.gov.ab.ca/$department/deptdocs.nsf/all/crop12127 (2010/12/05).

Hicks, K., Johnston, D. and A T. Hotchkiss, Jr. 2006. Increasing the Yield of Fuel Ethanol from Barley with β-Glucanases and β-Glucosidases.
http://services.bepress.com/cgi/viewcontent.cgi?article=1033&context=eci/bioenergy_i (2010/12/05)

Hill, B. and D.A. Pulkinen. 1988. A study of factors affecting pellet durability and pelleing efficiency in the production of dehydrated alfalfa pellets. A special report, *Saskatchewan Dehydrators Association*, Tisdale, SK, Canada: 25.

Johansson, L. S., Tullin. C, Leckner. B and P. Sjovall. 2003. Particle emissions from biomass combustion in small combustors. *Biomass and Bioenergy* 25(4):435–46.

Kaliyan, N., and R.V. Morey. 2006. Densification characteristics of corn stover and switchgrass. ASABE Paper No. 066174. St. Joseph, MI: ASABE.

Kaliyan, N. and R. V. Morey. 2008. Factors affecting strength and durability of densified biomass products. *Biomass and Bioenergy* 33:337-359.

Li, Y., and H. Liu. 2000. High-pressure densification of wood residues to form an upgraded fuel. *Biomass and Bioenergy* 19:177-186.

Mani, S., L.G. Tabil and S. Sokhansanj. 2002 . Grinding performance and physical properties of selected biomass. ASABE Paper No. 02-6175.Chicago, Illinois, USA: ASABE.

Mani, S., L.G. Tabil and S. Sokhansanj. 2004a. Grinding performance and physical properties of wheat and barley straws, corn stover and switchrgrass. *Biomass and Bioenergy*. 27:339-352.

Mani, S., L.G. Tabil and S. Sokhansanj. 2004b. Mechanical properties of corn stover grind. *American Society of Agricultural Engineers* 47(6):1983-1990.

Mani, S., L.G. Tabil and S. Sokhansanj. 2004c. Evaluation of compaction equations applied to four biomass species. *Canadian Biosystems Engineering* 46:3.55-3.61.

Mani, S., L.G. Tabil and S. Sokhansanj. 2006a. Specific energy requirement for compacting corn stover. *Bioresource Technology* 97:1420-1426.

Mani, S., L.G. Tabil and S. Sokhansanj. 2006b. Effects of compressive force, particle size and moisture content on mechanical properties of biomass pellets from grasses. *Biomass and Bioenergy* 30:648-654.

Mani, S., S. Sokhansanj, X. Bi and A. Thurlow. 2006c. Economics of producing fuel pellets from biomass. *Applied Engineering in Agriculture*. 22(3):421-426. ASABE.

Mohsenin, N. N. 1986. Friction. In *Physical Properties of Plant and Animal Materials*, Part II of Vol. I. *Texture of Foods, Mechanical Damage, Aero- and Hydrodynamic Characteristics and Frictional Properties*,. Department of Agricultural Engineering, The Pennsylvania State University. 636-642.

Molenda. M., Thompson. S.A., and I. J. Ross. 2000. Friction of Wheat on Corrugated and Smooth Galvanized Steel Surfaces. *Journal of Agricultural Engineering Resources*, 77 (2): 209-219.

Mozammel, H., S. Shahab, B. Tony, M. Sudhagar, J. Ladan, J. Lim and M. Afzal. 2006. Interaction of particle size, moisture content and compression pressure on the bulk density of wood chip and straw. ASABE Paper No. 06-100. St. Joseph, MI: ASABE.

OECD-FAO Agricultural Outlook 2010-2019. http://stats.oecd.org/viewhtml.aspx?QueryId=23342&vh=0000&vf=0&l&il=blank&lang=en (2010/12/02).

Olsson. M, Kjallstrand. J and G. Petersson. 2003. Specific chimney emissions and biofuel characteristics of softwood pellets for residential heating in Sweden. *Biomass and Bioenergy* 24:51-57.

Olsson, M. and J. Kjallstand. 2004. Emissions from burning of softwood pellets. *Biomass and Bioenergy* 27:607–611.

Olsson, M. 2006. Wheat straw and pea for fuel pellets-organic compounds from combustion. *Biomass and Bioenergy* 30:555-564.

Opoku, A., L.G. Tabil, and O. Fadeyi, 2009. Pelleting wheat based distiller's dried grains with solubles: quality characteristics. ASABE Paper No. 096665. Reno, Nevada:ASABE.

Orth, H.W. and R. Löwe. 1977. Influence of temperature on wafering in a continuous extrusion process. *Journal of Agricultural Engineering Research* 22(3):283-289.

Nuez-Ortín, W.G. and Yu. Peiqiang. 2009. Nutrient variation and availability of wheat DDGS, corn DDGS and blend DDGS from bioethanol plants. *Journal of the Science of Food and Agriculture.* 89:1754-1761.

Raghavan, J.K., and H.N. Conkle. 1991. Physical characteristic measurements for reconstituted coal pellets. *In Proceedings- Biennial Conference of the Institute for Briquetting and Agglomeration (IBA),* 22:85-96.

Rasco, B.A., F. M. Dong, A. E. Hashisaka, S. S. Gazzaz, S. E. Downey and M. L. S. Buenaventura. 1987. *Journal of Food Science* 52(1):236-237.

Rentsen, B. 2010. Factors affecting the quality of biomass fuel pellets. M.Sc. thesis. Saskatoon, SK.: University of Saskatchewan, Department of Agricultural and Bioresource Engineering.

Rhen. C., Gref. R., Sjostrom. M., and Wasterlund. 2005. Effects of raw material moisture content, densification pressure and temperature on some properties of Norway spruce pellets. *Fuel Processing Technology* 87:11-16.

Richards, S.R. 1990. Physical testing of fuel briquettes. *Fuel Processing Technology* 25:89-100.

Robinson, R. 1984. Pelleting. In *Manufacture of Animal feed*, ed. Beaven, D.A., 50-53. Herts, England: Turret-Wheatland Ltd.

Rosentrater, K.A. 2006. Some physical properties of distillers' dried grains with solubles (DDGS). *Applied Engineering in Agriculture.* 22(4):589-595.

Rosentrater, K.A. and Muthukumarappan, K. 2006. Corn ethanol co products: generation, properties, and future prospects. *International Sugar Journal* 108 (1295):648-657.

Rosentrater, K. A. 2007. Can you really pellet DDGS?. *Distillers Grains Quarterly.*

Rosentrater, K. A. 2008. Adding value to distillers grains by pelleting. USDA-ARS, North Central Agricultural Research Labaratory. Brookings, SD.

Rosentrater, Kurt. A and E. Kongar. 2009. Costs of pelleting to enhance the logistics of distillers' grains shipping. ASABE Paper No. BIO-097992. Seattle, WA: ASABE.

Seville, J. P. K., U. Tűzűn, and R. Clift. 1997. In *Processing of Particulate Solids*. London, U.K.: Chapman and Hall.

Shaw, M. and L. Tabil. 2006. Mechanical properties of selected biomass grinds. ASABE Paper No. 066175. St. Joseph, MI: ASABE.

Shaw, M. and L. Tabil. 2007. Compression and relaxation characteristics of selected biomass grinds. ASABE Paper No. 076183. Minneapolis, Minnesota: ASABE

Shaw, M. 2008. Feedstock and process variables influencing biomass densification. M.Sc thesis. Saskatoon, SK.: University of Saskatchewan, Department of Agricultural and Bioresource Engineering.

Shurson, J. and S. Noll. 2005. Feed Alternative Uses for DDGS. En-ergy from Agriculture: New Technologies, Innovative Pro-grams, and Success Stories. St. Louis, MO.

Skoch, E.R., K.C. Behnke, C.W. Deyoe and S.F. Binder. 1981. The effect of steam conditioning rate on the pelleting process. *Animal Feed Science and Technology* 6:83-90.

Singh, V., R.A. Moreau, K. B. Hicks, R.L. Belyea, and C.H. Staff. 2001. Removal of fibre from distillers' dried grains with solubles (DDGS) to increase value. *Transaction of the ASAE*. Vol 45(2): 389-392.

Sitkei, G. 1986. In *Mechanics of Agricultural Materials*. New York, NY: Elsevier Science Publishing Co., Inc.

Smith, I.E., S.D. Probert, R.E. Stokes and R.J. Hansford. 1977. The briquetting of wheat straw. *Journal of Agricultural Engineering Research.* 22:105-111.

Snyder, L. H., Roller.W.L and G. E. Hall. 1967. Coefficients of kinetic friction of wheat on various metal surfaces. Transactions of ASAE 10(3): 411-419.

Speihs, M. J., M .H. Whiney, and G.C. Shuron. 2002. Nutrient database for distillers' dried grains with solubles produced from new ethanol plants in Minnesota and South Dakota. *Journal of Animal Science*. Vol 80: 2639-2645.

Tabil, L.G., Jr. 1996. Binding and pelleting characteristics of alfalfa. PhD diss. Saskatoon, SK.: University of Saskatchewan, Department of Agricultural and Bioresource Engineering.

Tabil, L., Jr., and S. Sokhansanj. 1996. Process conditions affecting the physical quality of alfalfa pellets. *Applied Engineering in Agriculture* 12(3):345-350.

Tabil, L.G., Jr., and S. Sokhansanj. 1997. Bulk properties of alfalfa grind in relation to its compaction characteristics. *Applied Engineering in Agriculture* 13(4):499-505.

The Bioenergy Site. 2010. http://www.thebioenergysite.com/articles/664/biofuel-production-greater-shares-of-commodities-used (2010/12/02).

The mathworks- MATLAB and simulink for technical computing. 2010. Full factorial designs.http://www.mathworks.com/access/helpdesk/help/toolbox/stats/f7553.html (2010/08/20).

Thomas, M., and A.F.B. van der Poel. 1996. Physical quality of pelleted animal feed. Criteria for pellet quality. *Animal Feed Science Technology* 61:89-112.

Thompson .S .A., and I. J. Ross. 1983. Compressibility and frictional coefficients of wheat. *Transactions of the ASAE* 26(4):1171-1176.

Tsang-Mui-Chung, M., L.R. Verma and M.E. Wright. 1984. A device for friction measurement of grains. *Transactions of the ASAE* 27(6):1938-1941.

Tumuluru, J. S., L. Tabil, A. Opoku, M. Mosqueda, O. Fadeyi. 2010. Effect of process variables on the quality characteristics of pelleted wheat distiller's dried grains with solubles. *Biosystems Engineering*. 105:466-475.

Vest, L. 1993. Southeastern survey: factors which influence pellet production and quality. *Feed Management* 44(5):60-68.

Webb, P. A. 2001. Volume and Density Determinations for Particle Technologists. http://www.micromeritics.com/Repository/Files/density_determinations.pdf (2010/06/20).

Wheat DDGS. 2010. http://www.ddgs.usask.ca/ (2010/11/20)

Winowiski, T. 1998. Examining a new concept in measuring pellet quality: which test is best? *Feed Management* 49(1):23-26.

Wu, Y.V. and K. R. Sexson. 1984. Fractionation and characterization of protein-rich material from sorghum alcohol distillation. *Cereal Chemistry.* 61(5):388-391.

Xu, Y., M. A. Hanna, and R. Weber. 2008. Compaction of corn distillers dried grains. *Cereal Chemistry*. 85(2):158-164.

APPENDIX A

A.1 Analysis of variance of densities of ground DDGS (Noramera Bioenergy Corp.)

Dependent Variable: Bulk Density

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	102.838(a)	1	102.838	.140	.727
Intercept	2870693.340	1	2870693.340	3904.366	.000
Screen Size	102.838	1	102.838	.140	.727
Error	2941.009	4	735.252		
Total	2873737.186	6			
Corrected Total	3043.846	5			

R Squared = .834 (Adjusted R Squared = .808)

Dependent Variable: Particle Density

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	6337.500(a)	1	6337.500	12.294	.025
Intercept	10304461.500	1	10304461.500	19989.256	.000
Screen Size	6337.500	1	6337.500	12.294	.025
Error	2062.000	4	515.500		
Total	10312861.000	6			
Corrected Total	8399.500	5			

R Squared = .755 (Adjusted R Squared = .693)

A.2 Analysis of variance of densities of ground DDGS (Terra Grain Fuels Ltd.)

Dependent Variable: Bulk Density

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	3869.436(a)	1	3869.436	75.553	.001
Intercept	1554638.704	1	1554638.704	30355.352	.000
Screen size	3869.436	1	3869.436	75.553	.001
Error	204.859	4	51.215		
Total	1558712.999	6			
Corrected Total	4074.295	5			

a R Squared = .950 (Adjusted R Squared = .937)

Dependent Variable: Particle Density

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	4056.000(a)	1	4056.000	52.675	.002
Intercept	4263894.000	1	4263894.000	55375.247	.000
Screen size	4056.000	1	4056.000	52.675	.002
Error	308.000	4	77.000		
Total	4268258.000	6			
Corrected Total	4364.000	5			

a R Squared = .929 (Adjusted R Squared = .912)

A.3 Analysis of variance of durability of pellets produced from Noramera Bioenergy Corp.

SUMMARY

Groups	Count	Sum	Average	Variance
Column 1	24.0000	96.0000	4.0000	0.6678
Column 2	24.0000	354.0000	14.7500	0.5870
Column 3	24.0000	2280.0000	95.0000	26.0870
Column 4	24.0000	2043.0960	85.1290	32.3878

ANOVA

Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	158815.0795	3	52938.3598	3545.2055	0.0000	2.7036
Within Groups	1373.7791	92	14.9324			
Total	160188.8586	95				

A.4 Analysis of variance of durability of pellets produced from Terra Grain Fuels Ltd.

SUMMARY

Groups	Count	Sum	Average	Variance
Column 1	24.000	96.000	4.000	0.668
Column 2	24.000	295.080	12.295	0.660
Column 3	24.000	2280.000	95.000	26.087
Column 4	24.000	2099.299	87.471	54.331

ANOVA

Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	167192.314	3	55730.771	2727.034	0.000	2.704
Within Groups	1880.149	92	20.436			
Total	169072.464	95				

A.5 Analysis of variance of change in length, diameter, ratio of length/diameter of Noramera Bioenergy Corp. pellets.

Dependent Variable: Length

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	.870(a)	7	.124	49.651	.000
Intercept	4.430	1	4.430	1769.660	.000
Screen size	.000	1	.000	.162	.689
Moisture content	.803	1	.803	320.829	.000
Temperature	.001	1	.001	.508	.478
Screen size*Moisture content	.028	1	.028	11.383	.001
Screen size*Temperature	.019	1	.019	7.788	.007
Moisture content*Temperature	.003	1	.003	1.117	.294
Screen size*Moisture content*Temperature	.014	1	.014	5.769	.019
Error	.180	72	.003		
Total	5.481	80			
Corrected Total	1.050	79			

a R Squared = .828 (Adjusted R Squared = .812)

Dependent Variable: Diameter

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	.012(a)	7	.002	11.642	.000
Intercept	.133	1	.133	902.095	.000
Screen size	.004	1	.004	27.104	.000
Moisture content	.000	1	.000	2.460	.121
Temperature	.001	1	.001	5.780	.019
Screen size*Moisture content	4.18E-005	1	4.18E-005	.283	.596
Screen size*Temperature	.001	1	.001	7.057	.010
Moisture content*Temperature	.002	1	.002	12.506	.001
Screen size*Moisture content*Temperature	.004	1	.004	26.305	.000
Error	.011	72	.011		
Total	.156	80			
Corrected Total	.023	79			

a R Squared = .891 (Adjusted R Squared = .865)

Dependent Variable: Length/Diameter

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	.018(a)	7	.003	41.868	.000
Intercept	.027	1	.027	440.968	.000
Screen size	.001	1	.001	15.539	.000
Moisture content	.016	1	.016	253.455	.000
Temperature	.000	1	.000	5.521	.022
Screen size*Moisture content	.001	1	.001	14.783	.000
Screen size*Temperature	6.90E-005	1	6.90E-005	1.124	.293
Moisture content*Temperature	.000	1	.000	1.810	.183
Screen size*Moisture content*Temperature	5.17E-005	1	5.17E-005	.842	.362
Error	.004	72	6.14E-005		
Total	.050	80			
Corrected Total	.022	79			

a R Squared = .803 (Adjusted R Squared = .784)

A.6 Analysis of variance of change in length, diameter, ratio of length/diameter of Terra Grain Fuel Ltd. pellets

Dependent Variable: Length

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	1.383(a)	7	.198	155.445	.000
Intercept	4.077	1	4.077	3208.988	.000
Screen size	.009	1	.009	7.361	.008
Moisture content	1.366	1	1.366	1075.307	.000
Temperature	.002	1	.002	1.489	.226
Screen size*Moisture content	.001	1	.001	.891	.348
Screen size*Temperature	.003	1	.003	2.469	.120
Moisture content*Temperature	.000	1	.000	.196	.660
Screen size*Moisture content*Temperature	.001	1	.001	.405	.526
Error	.091	72	.001		
Total	5.552	80			
Corrected Total	1.474	79			

a R Squared = .938 (Adjusted R Squared = .932)

Dependent Variable: Diameter

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	.010(a)	7	.001	13.464	.000
Intercept	.091	1	.091	842.886	.000
Screen size	.000	1	.000	2.562	.114
Moisture content	.007	1	.007	65.234	.000
Temperature	1.34E-005	1	1.34E-005	.125	.725
Screen size*Moisture content	.002	1	.002	17.136	.000
Screen size*Temperature	2.29E-005	1	2.29E-005	.212	.647
Moisture content*Temperature	.001	1	.001	7.164	.009
Screen size*Moisture content*Temperature	.000	1	.000	1.814	.182
Error	.008	72	.008		
Total	.109	80			
Corrected Total	.018	79			

a R Squared = .667 (Adjusted R Squared = .625)

Dependent Variable: Length/Diameter

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	.021(a)	7	.003	82.070	.000
Intercept	.032	1	.032	893.809	.000
Screen size	.000	1	.000	12.689	.001
Moisture content	.020	1	.020	549.265	.000
Temperature	7.64E-005	1	7.64E-005	2.105	.151
Screen size*Moisture content	.000	1	.000	4.090	.047
Screen size*Temperature	4.74E-005	1	4.74E-005	1.306	.257
Moisture content*Temperature	8.74E-005	1	8.74E-005	2.406	.125
Screen size*Moisture content*Temperature	9.55E-005	1	9.55E-005	2.629	.109
Error	.003	72	3.63E-005		
Total	.056	80			
Corrected Total	.023	79			

a R Squared = .889 (Adjusted R Squared = .878)

A.7 Analysis of variance of bulk and particle density; and porosity of Noramera Bioenergy Corp. pellets

Bulk density: Bd; Particle density: Pd; Porosity: P

Source	Dependent Variable	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	Bd	33463.335(a)	7	4780.476	98.054	.000
	Pd	38870.443(b)	7	5552.920	27.990	.000
	P	207.729(c)	7	29.676	26.016	.000
Intercept	Bd	10194595.290	1	10194595.290	209104.158	.000
	Pd	27941956.640	1	27941956.640	140844.077	.000
	P	37591.827	1	37591.827	32956.268	.000
Screen size	Bd	7535.962	1	7535.962	154.572	.000
	Pd	35939.916	1	35939.916	181.159	.000
	P	6.772	1	6.772	5.937	.027
Moisture content	Bd	317.699	1	317.699	6.516	.021
	Pd	793.730	1	793.730	4.001	.063
	P	11.105	1	11.105	9.735	.007
Temperature	Bd	24918.948	1	24918.948	511.119	.000
	Pd	696.173	1	696.173	3.509	.079
	P	175.052	1	175.052	153.466	.000
Screen size*Moisture content	Bd	17.579	1	17.579	.361	.557
	Pd	350.829	1	350.829	1.768	.202
	P	2.645	1	2.645	2.319	.147
Screen size*Temperature	Bd	454.488	1	454.488	9.322	.008
	Pd	125.858	1	125.858	.634	.437
	P	3.363	1	3.363	2.948	.105
Moisture content*Temperature	Bd	216.721	1	216.721	4.445	.051
	Pd	415.002	1	415.002	2.092	.167
	P	6.980	1	6.980	6.120	.025
Screen size*Moisture content* Temperature	Bd	1.938	1	1.938	.040	.844
	Pd	548.935	1	548.935	2.767	.116
	P	1.811	1	1.811	1.588	.226
Error	Bd	780.059	16	48.754		
	Pd	3174.229	16	198.389		
	P	18.251	16	1.141		
Total	Bd	10228838.683	24			
	Pd	27984001.312	24			
	P	37817.806	24			
Corrected Total	Bd	34243.393	23			
	Pd	42044.672	23			
	P	225.979	23			

a R Squared = .977 (Adjusted R Squared = .967)
b R Squared = .925 (Adjusted R Squared = .891)
c R Squared = .919 (Adjusted R Squared = .884)

A.8 Analysis of variance of bulk and particle density; and porosity of Terra Grain Fuels Ltd. pellets

Bulk density: Bd; Particle density: Pd; Porosity: P

Source	Dependent Variable	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	Bd	21956.068(a)	7	3136.581	40.532	.000
	Pd	2721.583(b)	7	388.798	6.500	.001
	P	226.211(c)	7	32.316	39.203	.000
Intercept	Bd	7642292.329	1	7642292.329	98756.878	.000
	Pd	24128631.896	1	24128631.896	403372.189	.000
	P	45860.420	1	45860.420	55634.023	.000
Screen size	Bd	288.982	1	288.982	3.734	.071
	Pd	2529.912	1	2529.912	42.294	.000
	P	20.404	1	20.404	24.753	.000
Moisture content	Bd	11587.857	1	11587.857	149.743	.000
	Pd	8.532	1	8.532	.143	.711
	P	111.062	1	111.062	134.731	.000
Temperature	Bd	8054.205	1	8054.205	104.080	.000
	Pd	41.475	1	41.475	.693	.417
	P	74.000	1	74.000	89.770	.000
Screen size*Moisture content	Bd	4.611	1	4.611	.060	.810
	Pd	62.953	1	62.953	1.052	.320
	P	.304	1	.304	.369	.552
Screen size*Temperature	Bd	175.500	1	175.500	2.268	.152
	Pd	46.956	1	46.956	.785	.389
	P	3.164	1	3.164	3.839	.068
Moisture content*Temperature	Bd	971.045	1	971.045	12.548	.003
	Pd	23.030	1	23.030	.385	.544
	P	7.757	1	7.757	9.410	.007
Screen size*Moisture content*Temperature	Bd	873.868	1	873.868	11.292	.004
	Pd	8.724	1	8.724	.146	.708
	P	9.520	1	9.520	11.549	.004
Error	Bd	1238.159	16	77.385		
	Pd	957.077	16	59.817		
	P	13.189	16	.824		
Total	Bd	7665486.555	24			
	Pd	24132310.555	24			
	P	46099.820	24			
Corrected Total	Bd	23194.227	23			
	Pd	3678.660	23			
	P	239.400	23			

a R Squared = .947 (Adjusted R Squared = .923)
b R Squared = .740 (Adjusted R Squared = .626)
c R Squared = .945 (Adjusted R Squared = .921)

A.9 Analysis of variance of hardness of pellets from Noramera Bioenergy Corp.

Dependent Variable: Hardness

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	320434.250(a)	8	40054.281	21.396	.000
Intercept	3307067.994	1	3307067.994	1766.579	.000
Screen size	135.382	1	135.382	.072	.789
Moisture content	316244.903	1	316244.903	168.933	.000
Temperature	233.006	1	233.006	.124	.725
Screen size*Moisture content	17.011	1	17.011	.009	.924
Screen size*Temperature	55.628	1	55.628	.030	.864
Moisture content*Temperature	25.867	1	25.867	.014	.907
Screen size*Moisture content* Temperature	173.844	1	173.844	.093	.761
Error	134785.278	72	1872.018		
Total	18395253.928	81			
Corrected Total	455219.528	80			

a R Squared = .704 (Adjusted R Squared = .671)

A.10 Analysis of variance of hardness of pellets from Terra Grain Fuels Ltd.

Dependent Variable: Hardness

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	1173834.87(a)	7	167690.696	105.849	.000
Intercept	32416932.581	1	32416932.58	20462.09	.000
Screen size	9.793	1	9.793	.006	.938
Moisture content	1169057.076	1	1169057.076	737.928	.000
Temperature	1453.598	1	1453.598	.918	.341
Screen size*Moisture content	261.328	1	261.328	.165	.686
Screen size*Temperature	816.706	1	816.706	.516	.475
Moisture content*Temperature	1889.471	1	1889.471	1.193	.278
Screen size*Moisture content*Temperature	346.903	1	346.903	.219	.641
Error	114065.506	72	1584.243		
Total	33704832.962	80			
Corrected Total	1287900.381	79			

a R Squared = .911 (Adjusted R Squared = .903)

A.11 Analysis of variance of durability of pellets from Noramera Bioenergy Corp.

Dependent Variable: Durability

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	634.39	7	90.63	13.11	0.0000
Intercept	173925.70	1	173925.70	25157.17	0.0000
Screen size	312.99	1	312.99	45.27	0.0000
Moisture content	223.32	1	223.32	32.30	0.0000
Temperature	21.49	1	21.49	3.11	0.0970
Screen size*Moisture content	12.03	1	12.03	1.74	0.2057
Screen size*Temperature	42.85	1	42.85	6.20	0.0242
Moisture content*Temperature	21.49	1	21.49	3.11	0.0970
Screen size*Moisture content*Temperature	0.22	1	0.22	0.03	0.8611
Error	110.62	16	6.91		
Total	174670.70	24			
Corrected Total	745.00	23			

a R Squared = .852 (Adjusted R Squared = .787)

A.12 Analysis of variance of durability of pellets from Terra Grain Fuels Ltd.

Dependent Variable: Durability

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	1188.894(a)	7	169.842	44.574	.000
Intercept	183629.270	1	183629.270	48192.179	.000
Screen size	549.031	1	549.031	144.089	.000
Moisture content	250.583	1	250.583	65.764	.000
Temperature	229.093	1	229.093	60.124	.000
Screen size*Moisture content	81.660	1	81.660	21.431	.000
Screen size*Temperature	.637	1	.637	.167	.688
Moisture content*Temperature	70.487	1	70.487	18.499	.001
Screen size*Moisture content*Temperature	7.404	1	7.404	1.943	.182
Error	60.966	16	3.810		
Total	184879.130	24			
Corrected Total	1249.860	23			

a R Squared = .951 (Adjusted R Squared = .930)

A.13 Analysis of variance of moisture absorption of pellets from Noramera Bioenergy Corp.

Dependent Variable: Moisture absorption

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	16202.354(a)	7	2314.622	17.484	.000
Intercept	168010.234	1	168010.234	1269.080	.000
Screen size	24.788	1	24.788	.187	.671
Moisture content	16055.687	1	16055.687	121.278	.000
Temperature	46.206	1	46.206	.349	.563
Screen size*Moisture content	67.845	1	67.845	.512	.484
Screen size*Temperature	4.782	1	4.782	.036	.852
Moisture content*Temperature	2.416	1	2.416	.018	.894
Screen size*Moisture content*Temperature	.630	1	.630	.005	.946
Error	2118.199	16	132.387		
Total	186330.788	24			
Corrected Total	18320.554	23			

a R Squared = .884 (Adjusted R Squared = .834)

A.14 Analysis of variance of moisture absorption of pellets from Terra Grain Fuels Ltd.

Dependent Variable: Moisture absorption

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	9346.517(a)	7	1335.217	22.895	.000
Intercept	145766.723	1	145766.723	2499.441	.000
Screen size	8.069	1	8.069	.138	.715
Moisture content	9260.667	1	9260.667	158.791	.000
Temperature	6.427	1	6.427	.110	.744
Screen size*Moisture content	2.459	1	2.459	.042	.840
Screen size*Temperature	1.647	1	1.647	.028	.869
Moisture content*Temperature	15.858	1	15.858	.272	.609
Screen size*Moisture content*Temperature	51.390	1	51.390	.881	.362
Error	933.116	16	58.320		
Total	156046.356	24			
Corrected Total	10279.633	23			

a R Squared = .909 (Adjusted R Squared = .870)

A.15 Analysis of variance of ash content of DDGS pellets

SUMMARY

Groups	Count	Sum	Average	Variance
Column 1	6	85.77	14.295	1.74243
Column 2	6	19.2	3.2	2.36658E-31
Column 3	6	570	95	30
Column 4	6	32.741	5.45683	0.395502167

ANOVA

Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	34747.23185	3	11582.4106	1441.587537	1.57742E-23	3.0983
Within Groups	160.6896608	20	8.034483			
Total	34907.92151	23				

APPENDIX B

B.1 Data used to plot the shear stress and normal stress graph of Noramera Bioenergy Corp. samples

Normal Force, N	Shear Force, N	Area, m^2	Normal stress, kPa	Shear stress, kPa
100.9449	92.0547583	0.01	10.09449	9.20547583
100.9449	94.80951416	0.01	10.09449	9.480951416
100.9449	93.20257324	0.01	10.09449	9.320257324
201.0069	108.5832935	0.01	20.10069	10.85832935
201.0069	109.9606714	0.01	20.10069	10.99606714
201.0069	108.5832935	0.01	20.10069	10.85832935
302.3442	116.1588721	0.01	30.23442	11.61588721
302.3442	118.4041162	0.01	30.23442	11.84041162
302.3442	117.765813	0.01	30.23442	11.7765813
400.9347	121.354502	0.01	40.09347	12.1354502
400.9347	125.1118286	0.01	40.09347	12.51118286
400.9347	128.0961475	0.01	40.09347	12.80961475

B.2 B.1 Data used to plot the shear stress and normal stress graph of Terra Grain Fuels Ltd. samples

Normal Force, N	Shear Force, N	Area, m^2	Normal stress, kPa	Shear stress, kPa
100.9449	81.72442383	0.01	10.09449	8.172442383
100.9449	83.79049072	0.01	10.09449	8.379049072
100.9449	86.08612061	0.01	10.09449	8.608612061
201.0069	92.51388428	0.01	20.10069	9.251388428
201.0069	94.35038818	0.01	20.10069	9.435038818
201.0069	99.8598999	0.01	20.10069	9.98598999
302.3442	105.3694116	0.01	30.23442	10.53694116
302.3442	103.7624707	0.01	30.23442	10.37624707
302.3442	99.40077393	0.01	30.23442	9.940077393
400.9347	113.8632422	0.01	40.09347	11.38632422
400.9347	116.847561	0.01	40.09347	11.6847561
400.9347	114.5519312	0.01	40.09347	11.45519312